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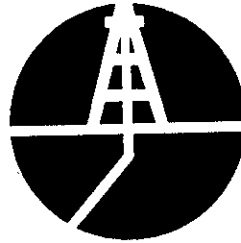
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DT-97-151

Cement Technology

November 1997

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## DEA 87 - PHASE II PROGRESS REPORT NO. 5

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Prepared for: DEA 87 Phase II Members  
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## **DEA 87 PHASE 2 PROGRESS REPORT NO. 5**

### **OBJECTIVE**

The objective of this project was to test and compare Blast Furnace Slag Cements with Portland Cement in two testing scenarios. The tests conducted were gas migration tests and annular sealing tests. This report summarizes the results of Phase II test data conducted on the Shell designed slag to cement slurry designs. These tests were the three Shell slurry designs that were lower density than previously tested slurries with non-progressive gel strengths.

### **SECTION I - GAS MIGRATION TESTS**

This section will outline the gas migration testing results obtained on the Shell Mix Formulations of PHPA Slag Cements. The gas migration test cell used in the testing is shown in schematic 1. The results for tests conducted are shown in plots 1a, b through 3a, b in the Appendix. In order for the data to be analyzed, each test result is shown in two plots. The first plot (plot a) shows the data in the first 1500 to 1600 minutes. The next plot (plot b) shows the data collected during the entire test time (2 to 7 days).

#### **Testing Procedures for Seven Day Tests:**

Testing conditions were as follows:

Bottom Hole Circulating Temperature (BHCT) = 126°F

Bottom Hole Static Temperature (BHST) = 152°F

8000 Ft Casing Job with 0.9°F/100 ft Gradient

The mud compositions used in the testing was as follows:

Nondispersed System (PHPA)
Sea Water
10.0 ppb Prehydrated Bentonite
1.5 ppb PHPA
2.0 ppb CMS
0.5 ppb PAC-L
NaOH to pH 9.5
Barite to 12.5 ppg
30.0 ppb Rev Dust
Density = 12.5 #/gal
Fluid Loss = 3.6 cc's/30 min.

#### The Shell Mix Formulations:

Shell Mix #1: PHPA (12.5 ppg diluted to 10.5 ppg) + 5.52 lbs/bbl Sodium Hydroxide (NaOH) + 13.81 lbs/bbl Sodium Tripoly Phosphate ( $\text{Na}_5\text{P}_3\text{O}_{10}$ ) + 314.3 ppb Blast Furnace Slag

Shell Mix #2: PHPA (12.5 ppg diluted to 10.5 ppg) + 4.43 lbs/bbl Sodium Hydroxide (NaOH) + 11.07 lbs/bbl Lime + 1.85 lbs/bbl Dispersant + 314.3 ppb Blast Furnace Slag

Shell Mix #3: PHPA (12.5 ppg diluted to 10.5 ppg) + 4.43 lbs/bbl Sodium Hydroxide (NaOH) + 11.07 lbs/bbl Sodium Carbonate ( $\text{Na}_2\text{CO}_3$ ) + 2 gal/bbl NRJ1428 Resin + 314.3 ppb Blast Furnace Slag

The Final Cementing Density was  $\approx 15.2$  lbs/gal for all three compositions.

The test procedures for conducting the gas migration tests were as follows:

1. Fill the model up with water and heat it to the BHCT.
2. Mix approximately 4 gal of the test slurry in a 5 gal bucket.
3. Heat the slurry up to the BHCT while continuously stirring the slurry under low shear.
4. Condition the slurries for one hour.
5. Pump the slurry into the model until good returns from the top of the model are observed.
6. Open valves for water column on top of the cement and gas pressure.
7. Maintain 1.7 psi differential between hydrostatic pressure and the gas pressure.
8. Increase Temperature of the model from BHCT to BHST.
9. Maintain the BHST for the entire testing time.
10. Record the following data:
  - a. Temperature of the Model
  - b. Pore pressure at several locations in the model (one at the gas entry point, and one at 6 feet from the bottom).
  - c. Amount of Gas entering the model (first 48 hours).
  - d. Amount of Gas flowing out of the model
11. At the end of the testing period evaluate the path of gas flow

## Laboratory Test Results

The laboratory design test results on each of the Shell Mix Formulations tested with the PHPA mud is shown in Table 1.

All the slurries had similar properties. The data for the Portland Cement system is shown for comparison purposes only. Thickening time and Ultrasonic Cement Analyzer (UCA) charts are in the Appendix at the end of this report.

**Table 1**  
**Slurry Test Data**

Slurry	Portland	Shell Mix #1	Shell Mix #2	Shell Mix #3
Thick Time (Hrs:min)	2:11	3:07	3:01	2:12
Free Fluid @ 80°F (% by vol.)	0	0	0	0
Free Fluid @ 126°F (% by vol.)	0	0	0	0
Fluid Loss (cc's / 30 min.)	69	334	242	228
UCA (50 psi) (500 psi) (24 hrs)	5:04 6:36 2820	4:36 5:02 1800	2:54 3:58 950	2:30 4:02 1071
Rheology (600 rpm) (300 rpm) Gel Str.) (10':10")	300+ 164  5: 17	103 60  13 : 15	139 67  4 : 25	300+ 165  55 : 52

**Gas Migration Test Results:**

Below is the summary of the tests conducted and a description of the data generated for each test. All of the plots are in the Appendix at the end of this report.

- Test / Shell Mix #1 - Plot #1a, b

Composition: PHPA Mud diluted from 12.5 ppg to 10.5 ppg, 5.52 ppb Sodium Hydroxide, 13.81 ppb Sodium Tripoly Phosphate, 314.3 ppb Blast Furnace Slag, Final Density of 15.22 ppg.

Test Comments: From plot 1a the gas entry started at 88 minutes and the gas out of the model was observed at 218 minutes. The gas flow continued for the entire 7 day testing period. A total of over 1800 cc's of gas entered the model. This is the maximum amount of gas that the gas entry set up can deliver. Plot 2b shows that a total gas out about 74300 cc was observed throughout the testing period. The Top Heise developed mechanical problems and quit reading data to the computer shortly after 200 minutes.

- Test / Shell Mix #2- Plot #2a, b

Composition: PHPA Mud diluted from 12.5 ppg to 10.5 ppg, 4.43 ppb Sodium Hydroxide, 11.07 ppb Lime, 1.85 ppb Dispersant (Unical), 314.3 ppb Blast Furnace Slag, Final Density of 15.17 ppg.

Test Comments: Plot 2a shows that the gas entry and exit from the model started about 400 minutes continued for the entire 7 day testing period. Plot 2b shows that a total of about 800 cc's of gas was measured out of the model during the entire 7 day testing program.

- Test / Shell Mix #3 - Plot #3a, b

Composition: PHPA Mud diluted from 12.5 ppg to 10.5 ppg, 4.43 ppb Sodium Hydroxide, 11.07 ppb Sodium Carbonate, 2 gal/bb NRJ1428 Resin, 314.3 ppb Blast Furnace Slag, Final Density of 15.13 ppg.

Test Comments: In this case, plot 3a shows that the gas entry and exit from the model was just before 200 minutes. Plot 3b indicates that only 68 cc's of gas was measured out of the model during the entire testing period. The pressures indicated a sharp increase at 7500 minutes.

### Permeability Measurements:

Tables 2, 3, and 4 summarizes the bulk permeability to gas that was observed and calculated in each of the tests. After the test models completed the seven day (just over 2 days for Shell Mix #1) testing period, the flow rate was measured at the top of the models. With the BHST being maintained the models were cut at one foot intervals starting from the top. A special adapter was made to capture the cross-sectional flow of gas through the model at each cut location so flow rate could be measured. Equation 1 was used to calculate the bulk permeability.

Equation 1:

$$k = (q_{sc} P_{sg} T Z \mu L) \sqrt{3.164 T_{sg} A (P_1^2 - P_2^2)}$$

where:

k = Permeability, darcy

$q_{sc}$  = Volumetric Flow rate of Gas, SCF/Day

$P_{sg}$  = Standard Pressure, psia

T = Temperature, °R

Z = Z factor

$\mu$  = Viscosity, cp

L = Length, ft

$T_{sg}$  = Standard Temperature, °R

A = Flowing Area, ft<sup>2</sup>

$P_1$  = High pressure, psi

$P_2$  = Low pressure, psi

The flow rate q is taken from the inlet gas volumetric flow rate and then adjusted to standard temperature and pressure. These measurements were taken with the model at the test temperature at 5 minute intervals up to 10 minutes.

The matrix permeability was low but the bulk permeability was high because of the flow of gas through the micro channel at the cement / pipe interface.

**Table 2**  
**Summary of Flow Rate & Bulk Permeability to Gas**  
**Shell Mix #1**

Photograph Cut	Length from Gas Inlet (ft)	Pressure (psi)	Flow Rate (cc's / min)	Perm (md)
	10	7.54	62.4	684
A	9	7.55	70.0	690
B	8	31.76	108.6	137
C	7	24.37	65.6	107
D	6	24.38	376	527
E	5	24.31	740.4	868
F	4	24.25	1124	1059
G	3	24.19	1694	1201
H	2	24.07	2898	1379
I	1	7.43	971	1084

**Table 3**  
**Summary of Flow Rate & Bulk Permeability to Gas**  
**Shell Mix #2**



Photograph Cut	Length from Gas Inlet (ft)	Pressure (psi)	Flow Rate (cc s/min)	Perm (md)
	10	31.3	0.1	0.21
A	9	31.3	0.1	0.10
B	8	31.3	0.0	0.00
C	7	31.3	0.0	0.00
D	6	31.3	0.1	0.06
E	5	31.3	0.2	0.16
F	4	31.3	1.5	0.95
G	3	31.3	2.4	1.16
H	2	31.3	8.0	2.58
I	1	31.3	568	91.6

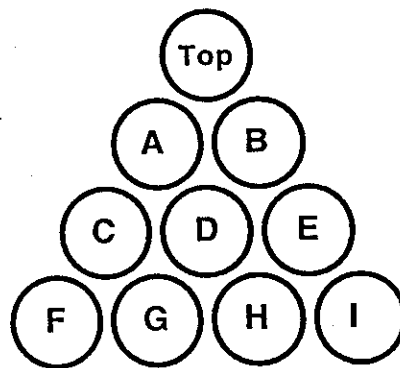
Table 4  
Summary of Flow Rate & Bulk Permeability to Gas  
Shell Mix #3

Photograph Cut	Length from Gas Inlet (ft)	Pressure (psi)	Flow Rate (cc's/min)	Perm (md)
	10	31.09	8.4	13.68
A	9	31.11	25.8	37.78
B	8	30.86	46.8	61.66
C	7	31.25	62.0	70.13
D	6	31.24	91.8	89.04
E	5	31.22	103.6	83.82
F	4	31.23	104.2	67.41
G	3	31.21	115.4	56.05
H	2	31.21	124.0	40.15
I	1	31.17	320.0	51.91

### Mapping Gas Flow Channels in Models

The model was cut in one foot intervals from the top down. Photos of the cuts of each gas migration tests are shown in figures 1 through 3 in the Appendix. The top cut is identified as Cut A down through Cut I on the bottom. The nitrogen pressure applied to the bottom of the model throughout the gas migration test was continuously maintained through out the cutting process. The temperature on the model was maintained at 152°F BHST through out the cutting process. Immediately after the cut had been made the exposed facial surface was wetted with soapy water. Any gas through the model was identified by the formation of bubbles. If the gas was from between the inner pipe wall and the outer cement surface the area was marked with a grease pen enclosing the area of bubbles. If the gas was coming through the matrix of cement the area was circled.

The group photo is laid out in the following pattern. A straight line was draw down the outside of the model, the model was cut in one foot sections and each section was orientated in the same position as cut from the model and photographed.



## SECTION 2 - DIMENSIONAL STABILITY

**OBJECTIVE**

The objective of this portion of Phase II is to measure the plastic state shrinkage, total volume reduction and the "gas tightness" of BFS and Portland cement systems by way of the Cement Hydration Analyzer. No slurries were tested in this section. It was recommended by the committee members that due to the limited data obtained in Phase I with this testing that no work would be performed in Phase II.

## SECTION 3 - ANNULAR SEALING TEST

### OBJECTIVE

The objective of this portion of the project was to test the ability of various BFS systems, and Portland Cement systems, to seal gas in an annular configuration. Well conditions simulating high stress was modeled. The flow rate of dry gas was measured in a full scale annular seal model containing several different slurries for periods up to 28 days. Additionally, the same models will investigate the ability of the systems to seal gas flow in the annulus under high stress conditions.

The tested composition was the following:

- Portland Class H cement + 1.0% Fluid Loss Additive + Bentonite + Dispersant at a Density of 15.6 #/gal  
Bentonite was used to meet design criteria for the cement slurry.
- The Shell Mix BFS slurries tested in this section were Shell Mix #2 and Shell Mix #3.

### Test Models

The test models are a 2 3/8" pipe (1.90" inside diameter ) inside a 5" pipe (4" inside diameter) 2 1/2' long (see schematic 2).

### Test Procedures

1. The slurries will be mixed in a one gallon mixer at room temperature.
2. The models annulus will be filled with slurry and placed in an oven and heated to BHST.
3. After curing for a period of over 72 hours the pressure stress test will be conducted.
  - a. Stress inside of center casing to preset pressure value for 5 minutes
  - b. Release the pressure inside the center casing and measure the flow rate of gas through the models cemented annulus with 50 psi gas pressure for 5 minutes.
  - c. Increase the pressure inside the center casing and repeat steps a and b.
  - d. Repeat steps c up to safety pressure limit of inside center casing.

Table 5, 6 and 7 summarizes the data for the slurries tested.

### **Measurement of Bulk Permeability**

The bulk permeability of the models were calculated from the flow data using Equation 1. Table 5 and Table 6 indicates that the Portland cement and the Shell Mix #2 system maintained a tight seal to gas even when after pressures up to 10,000 psi was placed on the inside of center casing for five minutes and then released. Table 7 indicated that the Shell Mix #3 system also maintained a tight seal to gas until 28 days at which time measurement showed a substantial amount of gas to flow relative to earlier measurements. The calculated bulk permeability however did not change much with increasing stress on the inside of the center casing.

After the test period was concluded each model was cut in half and gas flow through the cemented annulus was reestablished. The gas flow path was marked on the cut surface of the model which was then photographed. The photographs of each cut model are shown in figures 4, 5, and 6 in the Appendix.

Table 5  
Portland Cement System

Pressure of Gas (psi)	Time (days)	Pressure inside casing (psi)	Flow rate (cc's / min)	Perm (md)
50	4	0	15.6	0.83
50	4	1000	25.8	1.37
50	4	2000	28.0	1.48
50	4	3000	29.4	1.56
50	4	4000	27.2	1.44
50	4	5000	29.8	1.58
50	4	6000	30.2	1.60
50	4	7000	32.0	1.69
50	4	8000	30.4	1.61
50	4	9000	35.6	1.88
50	4	10000	38.4	2.03
50	14	0	14.0	0.74
50	14	10000	32.0	1.69
50	21	0	23.6	1.25
50	21	10000	39.4	2.09
50	28	0	26.2	1.39
50	28	10000	29.8	1.58

Table 6  
Shell Mix #2

Pressure of Gas (psi)	Time (days)	Pressure inside casing (psi)	Flow rate (cc's / min)	Perm (md)
50	4	0	2.9	0.16
50	4	1000	7.4	0.36
50	4	2000	6.6	0.35
50	4	3000	6.9	0.36
50	4	4000	5.8	0.31
50	4	5000	12.6	0.67
50	4	6000	18.8	0.99
50	4	7000	18.6	0.98
50	4	8000	22.2	1.17
50	4	9000	41.0	2.17
50	4	10000	51.8	2.74
50	14	0	21.0	1.11
50	14	10000	36.4	1.93
50	21	0	10.8	0.57
50	21	10000	54.2	2.87
50	28	0	62.1	3.29
50	28	10000	24.0	1.27

**Table 7**  
**Shell Mix #3**

Pressure of Gas (psi)	Time (days)	Pressure inside casing (psi)	Flow rate (cc's / min)	Perm (md)
50	4	0	1638	87
50	4	1000	2076	110
50	4	2000	2362	125
50	4	3000	2203	117
50	4	4000	2648	140
50	4	5000	2588	137
50	4	6000	2610	138
50	4	7000	2740	145
50	4	8000	3192	169
50	4	9000	3150	167
50	4	10000	2680	142
50	14	0	370	20
50	14	10000	2246	119
50	21	0	316	17
50	21	10000	1050	56
50	28	0	22440	1188
50	28	10000	24500	1297



# APPENDIX

## **Appendix**

**Schematic 1 - Gas Migration Model**

**Schematic 2 - Annular Seal Model**

**Plot 1 a - Gas Migration Test With Shell Mix #1 System (Short Time)**

**Plot 1 b - Gas Migration Test With Shell Mix #1 System (Entire Test Time)**

**Plot 2 a - Gas Migration Test With Shell Mix #2 System (Short Time)**

**Plot 2 b - Gas Migration Test With Shell Mix #2 System (Entire Test Time)**

**Plot 3 a - Gas Migration Test With Shell Mix #3 System (Short Time)**

**Plot 3 b - Gas Migration Test With Shell Mix #3 System (Entire Test Time)**

**Figure 1a-j: Cross Section Cuts of Gas Migration Model (Shell Mix #1 System)**

**Figure 2a-j: Cross Section Cuts of Gas Migration Model (Shell Mix #2 System)**

**Figure 3a-j: Cross Section Cuts of Gas Migration Model (Shell Mix #3 System)**

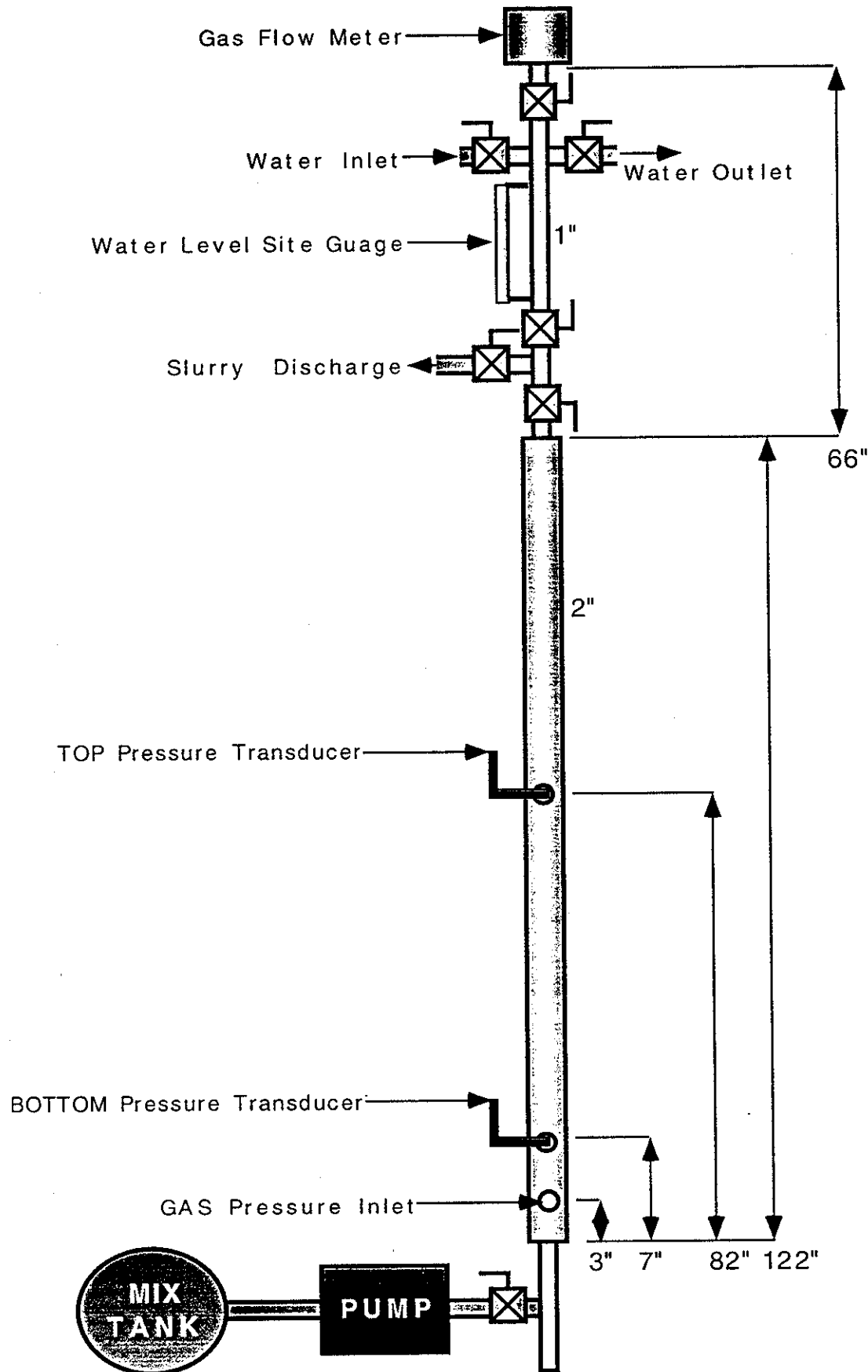
**Figure 4 - Cross Section Cut of Annular Sealing Model (Portland Cement)**

**Figure 5 - Cross Section Cut of Annular Sealing Model (Shell Mix #2 System)**

**Figure 6 - Cross Section Cut of Annular Sealing Model (Shell Mix #3 System)**

**Thickening Time and Ultrasonic Cement Analyzer charts**

Schematic 1  
DEA-87 PHASE II  
**GAS MIGRATION MODEL**

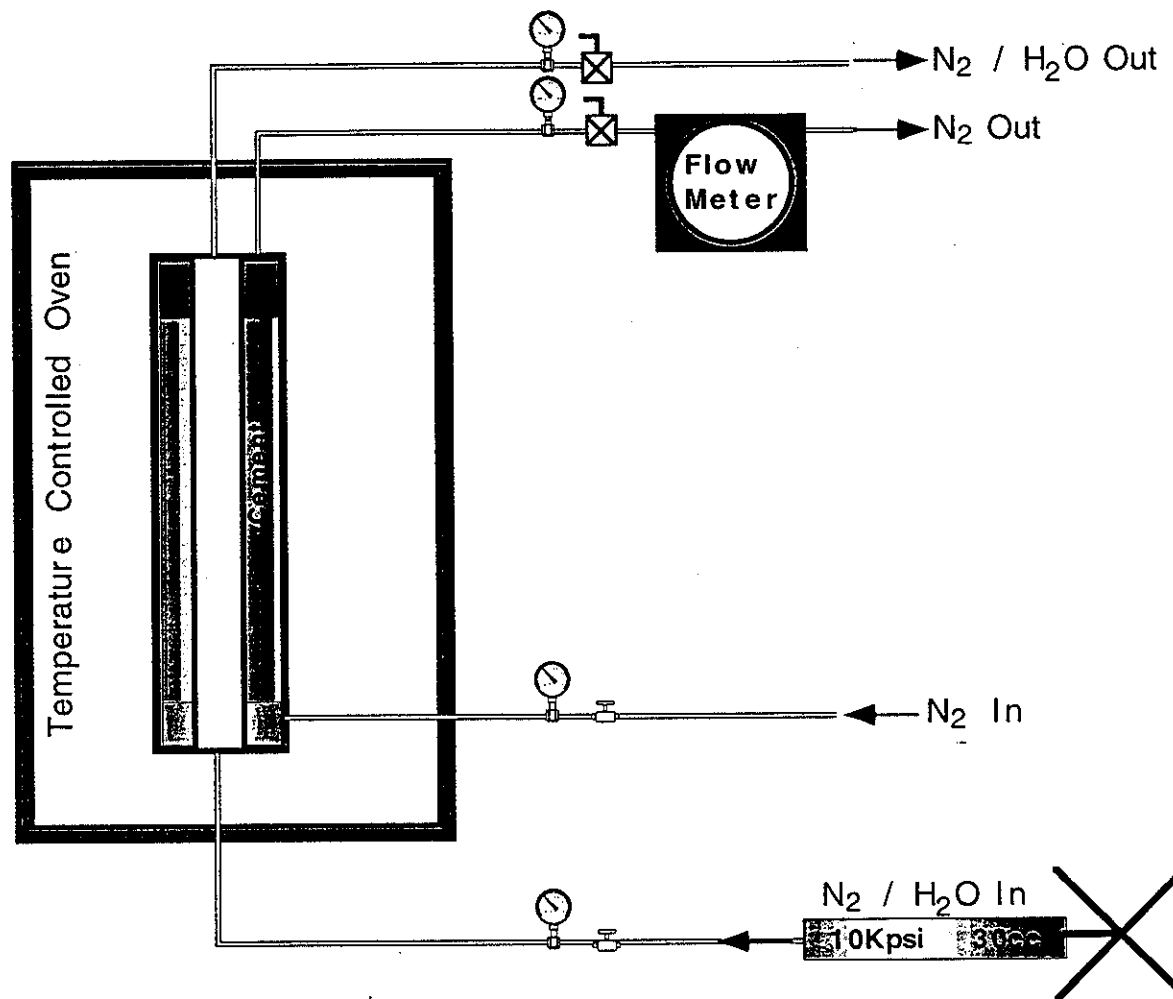


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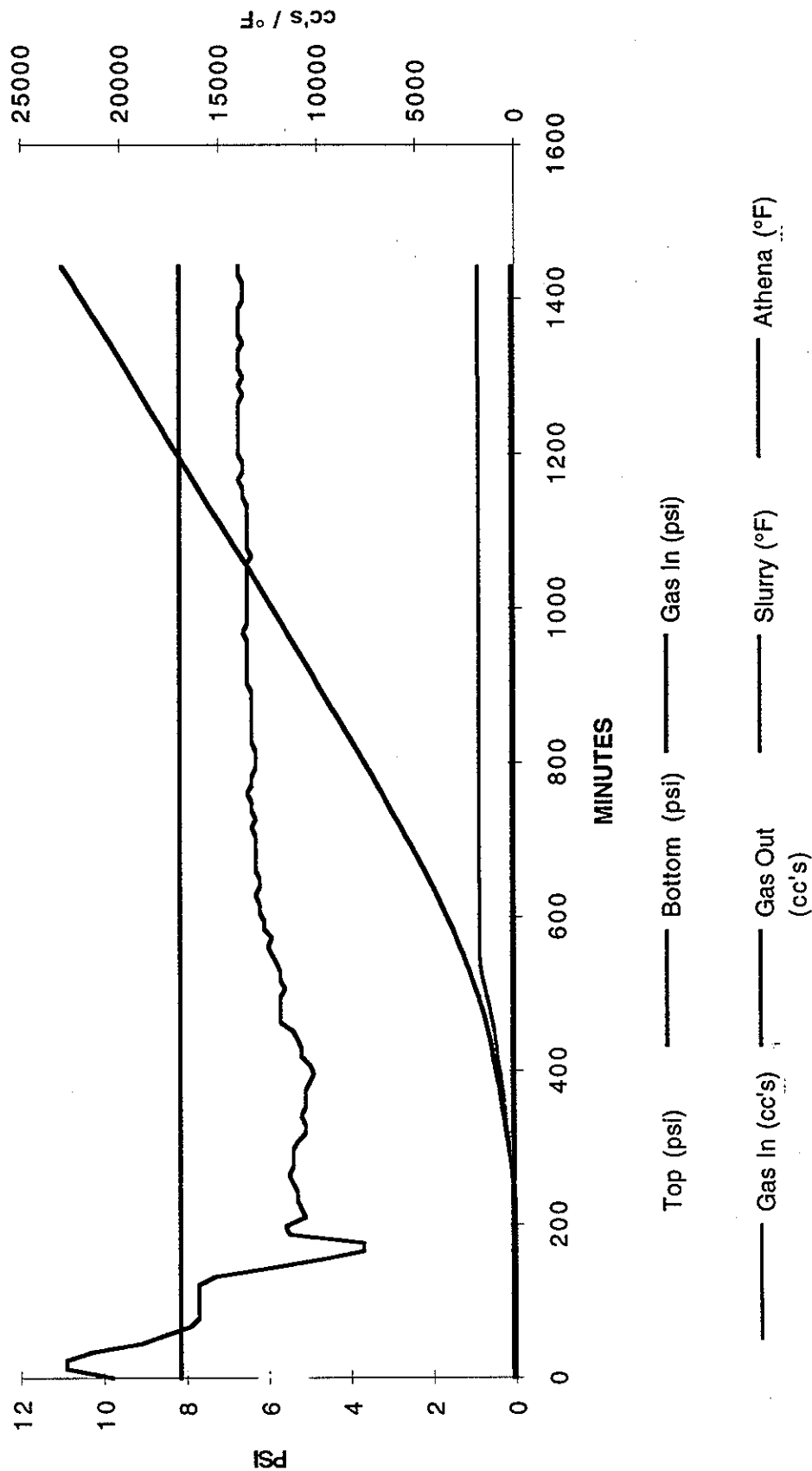


Schematic 2

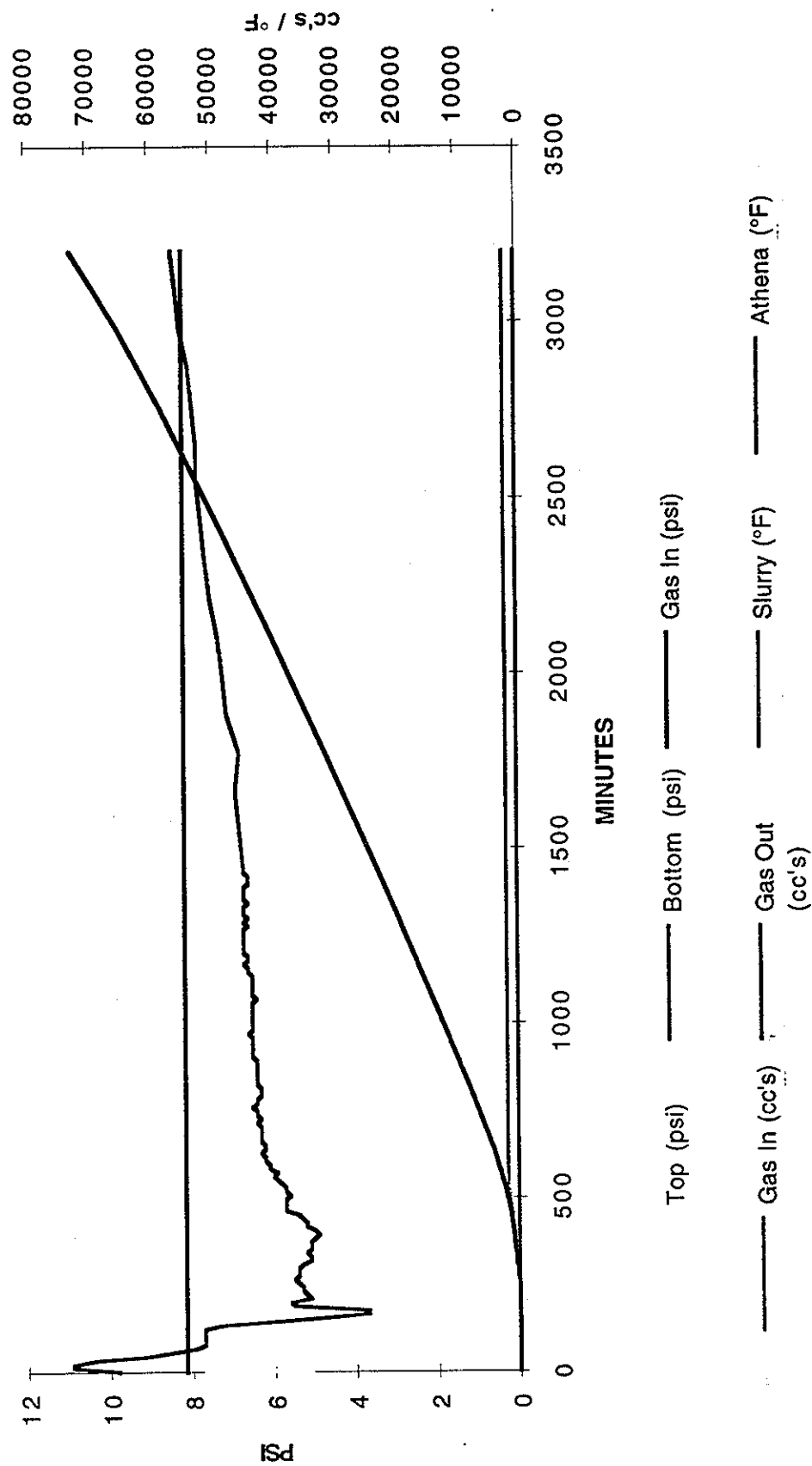
# ANNULAR SEALING TEST SCHEMATIC



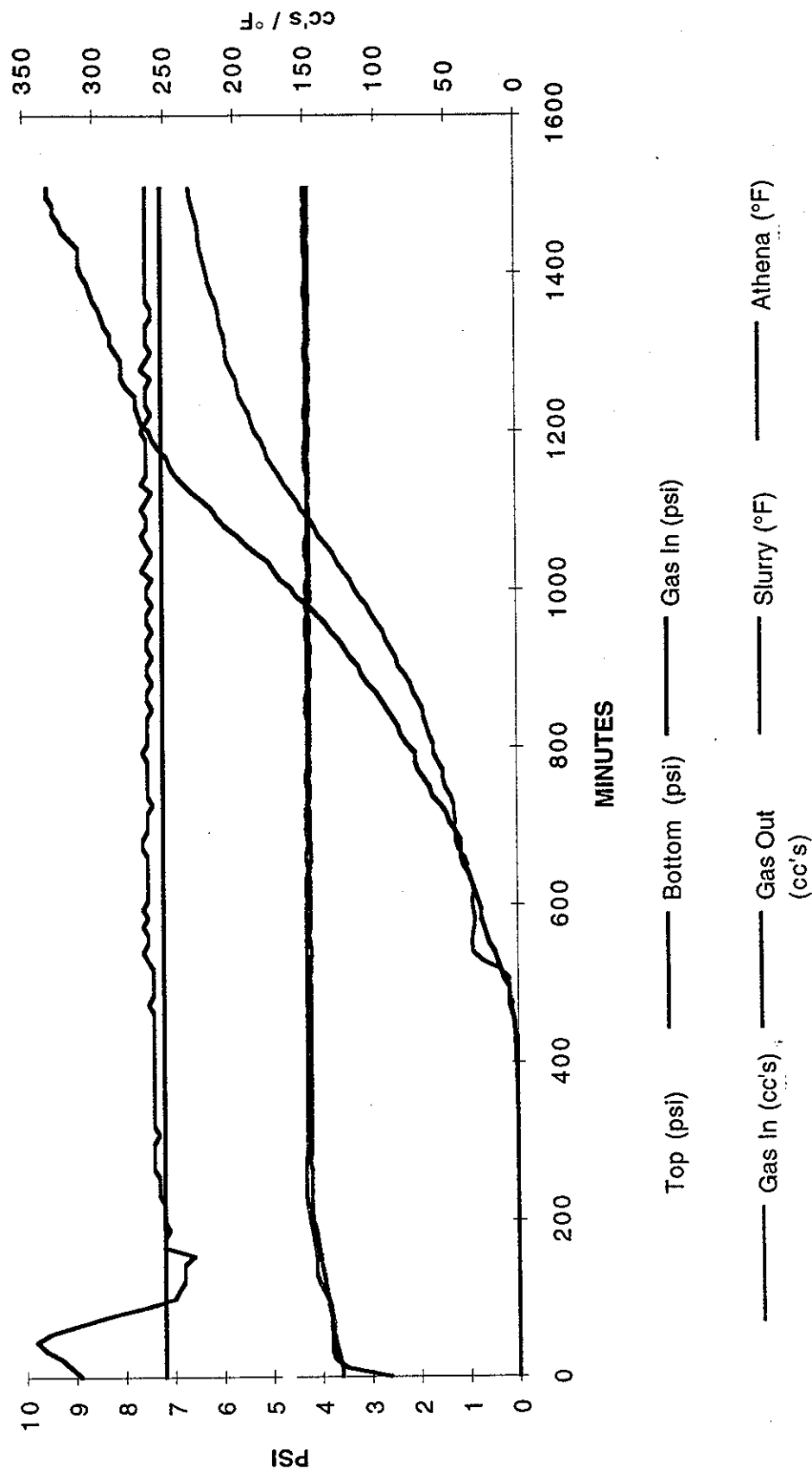
PHPA + 5.52 lbs/bbl Sodium Hydroxide + 13.81 lbs/bbl Sodium Tripoly Phosphate  
@ 15.2 ppg



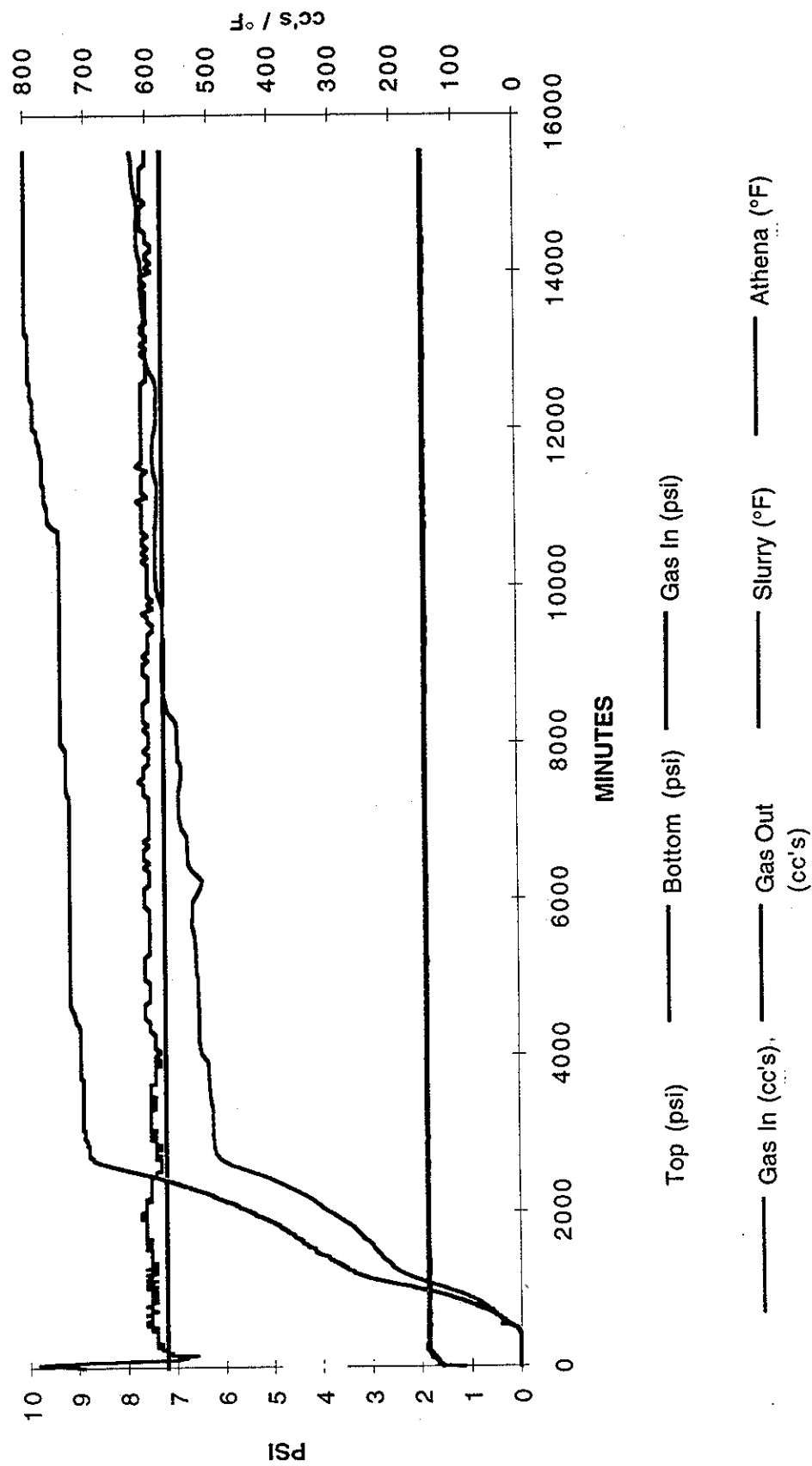
PHPA + 5.52 lbs/bbl Sodium Hydroxide + 13.81 lbs/bbl Sodium Tripoly Phosphate  
@ 15.2 ppb



PHPH + 4.43 lbs/bbl Sodium Hydroxide + 11.07 lbs/bbl Lime + 1.85 lbs/bbl  
Dispersant @ 15.2 ppg

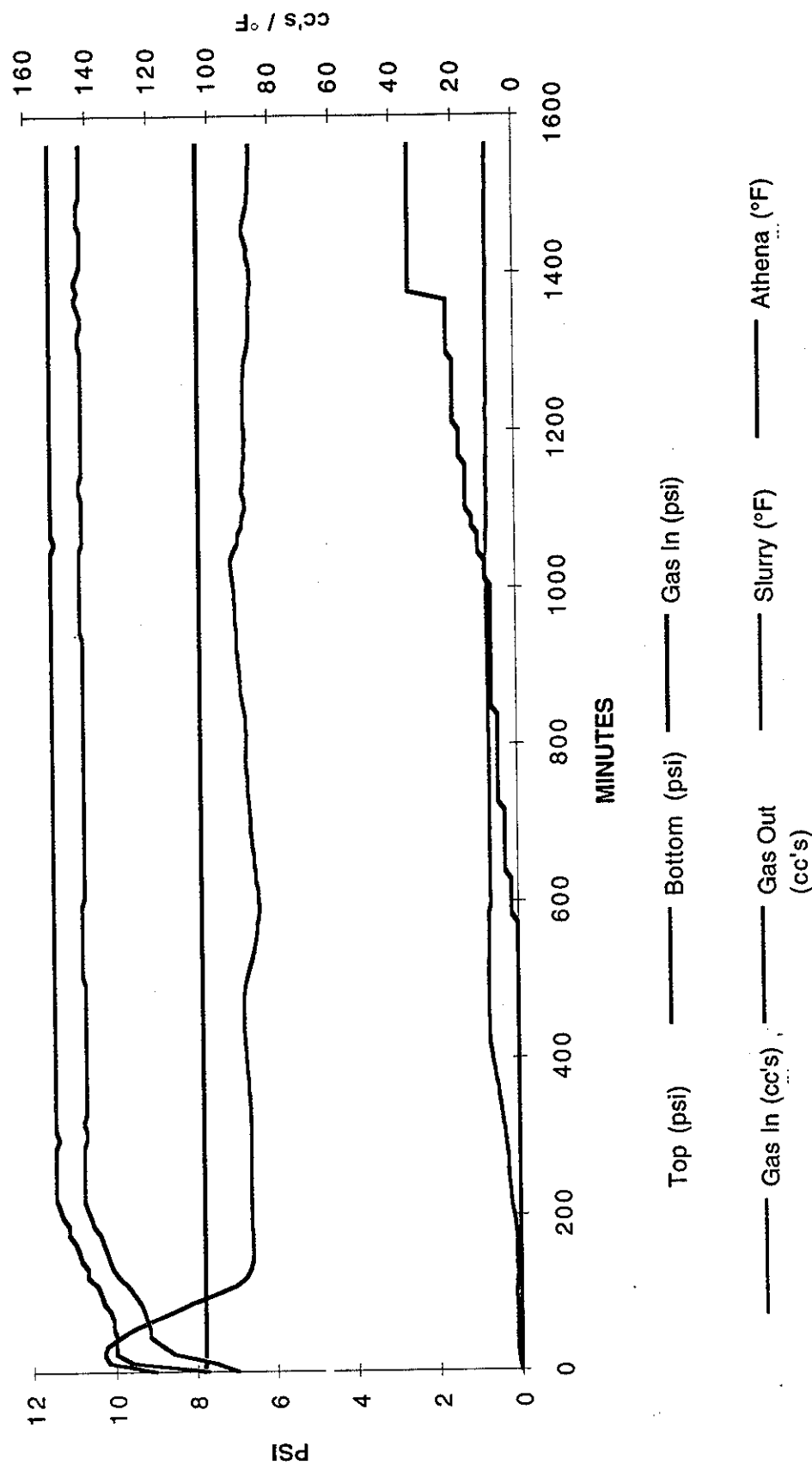


PHPA + 4.43 lbs/bbl Sodium Hydroxide + 11.07 lbs/bbl Lime + 1.85 lbs/bbl  
Dispersant @ 15.2 ppb

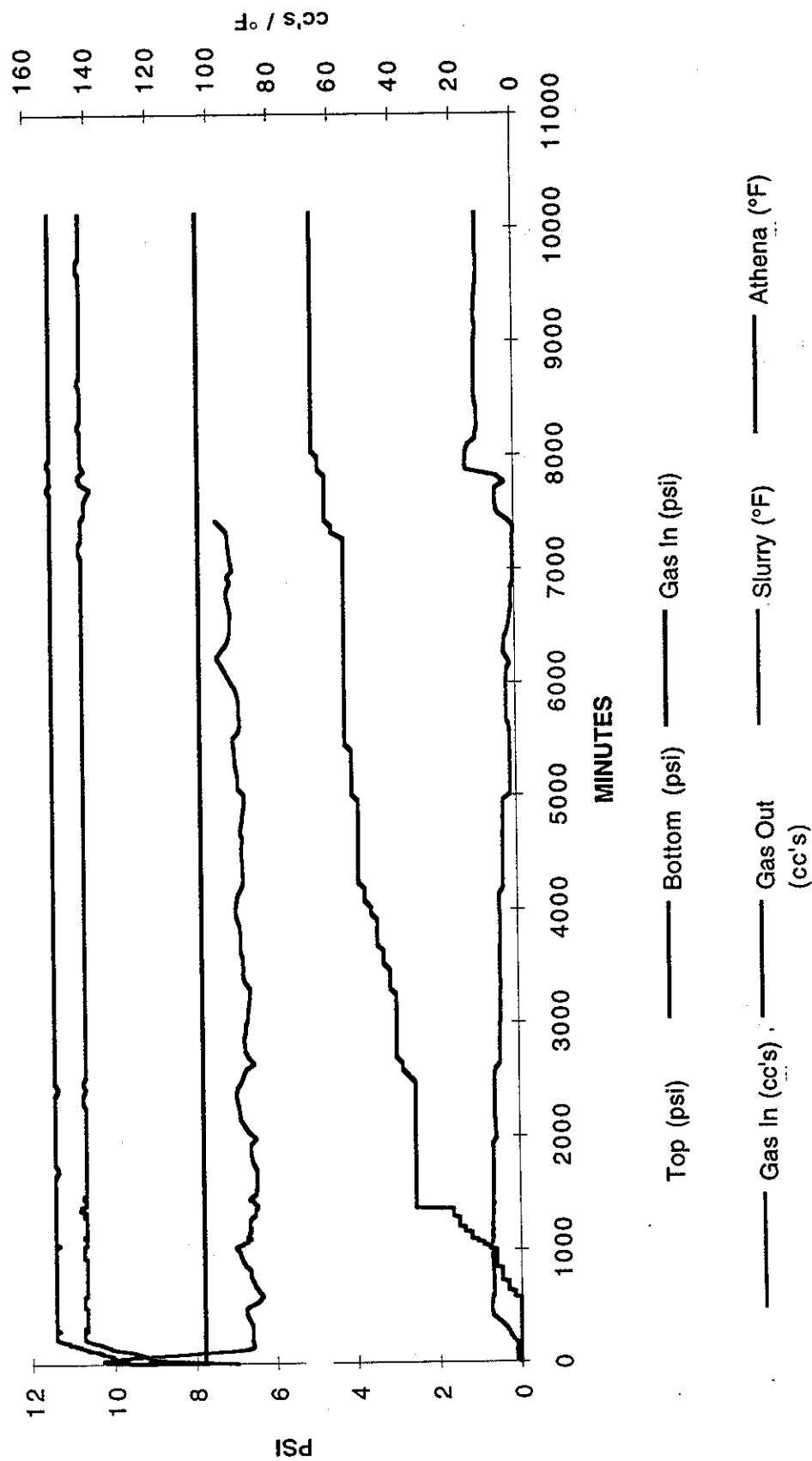




PHPA + 4.43 lbs/bbl Sodium Hydroxide + 11.07 lbs/bbl Sodium Carbonate + 2  
gals/bbl NRJ-1428 Resin @ 15.2 ppg



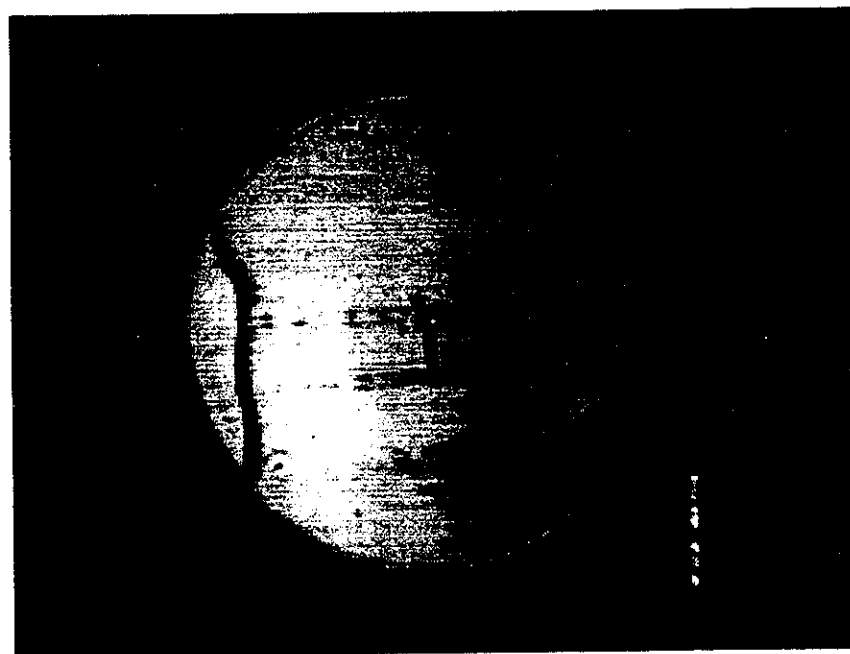
PHPA + 4.43 lbs/bbl Sodium Hydroxide + 13.81 lbs/bbl Sodium Carbonate + 2  
gal/bbl NPJ-1428 Resin @ 15.2 ppb





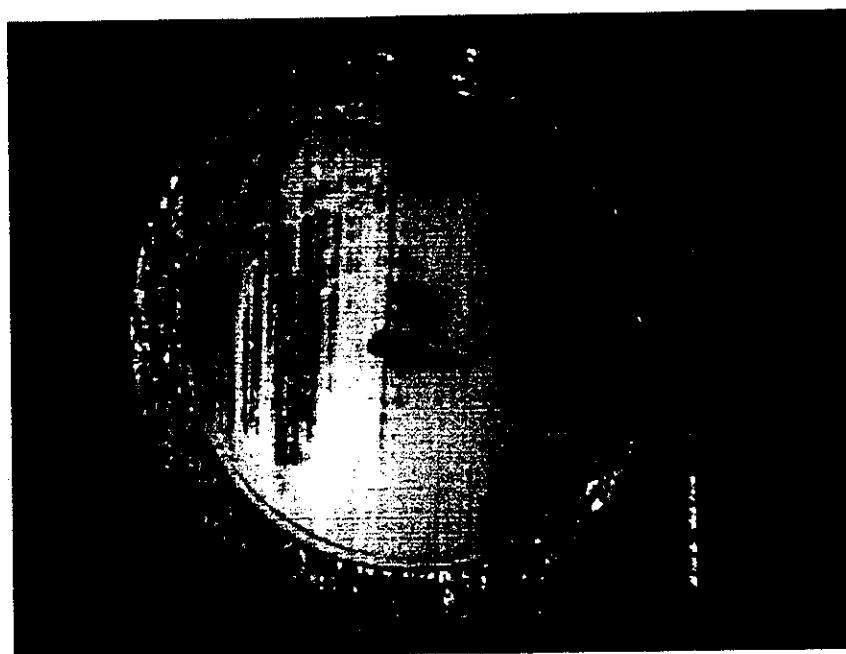
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**Figure 1a: Shell Mix #1 - Cut A**



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**Figure 1b: Shell Mix #1 - Cut B**



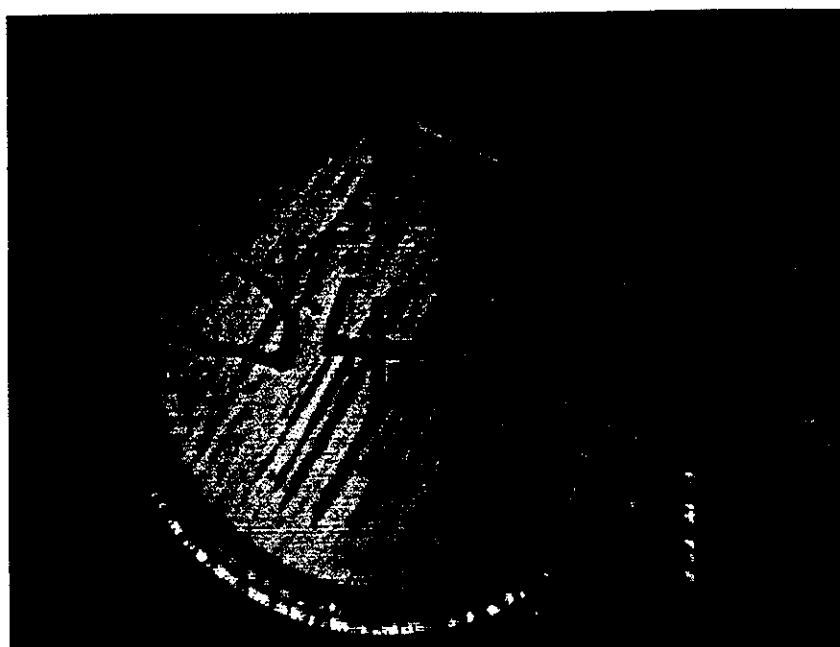
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Figure 1c: Shell Mix #1 - Cut C



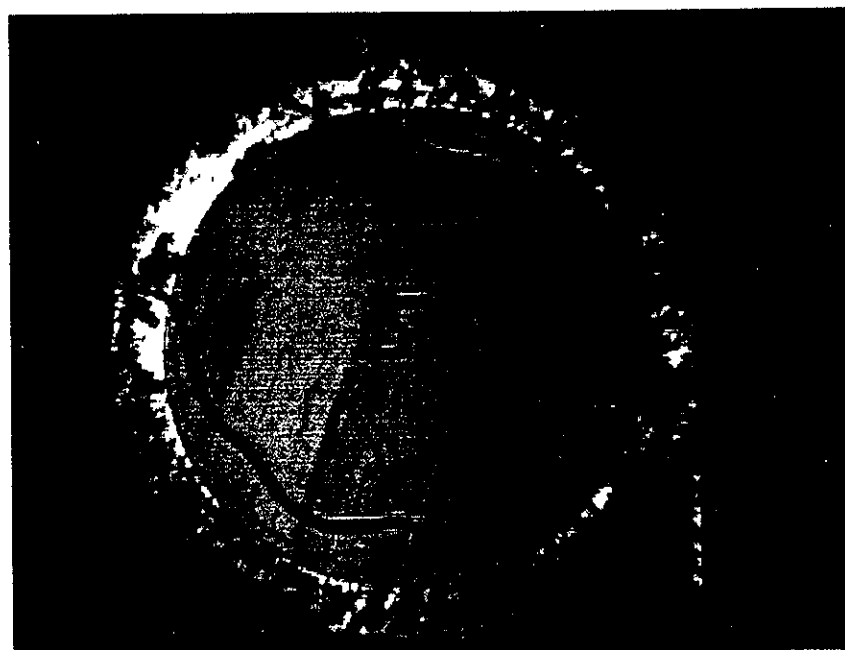
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Figure 1d: Shell Mix #1 - Cut D



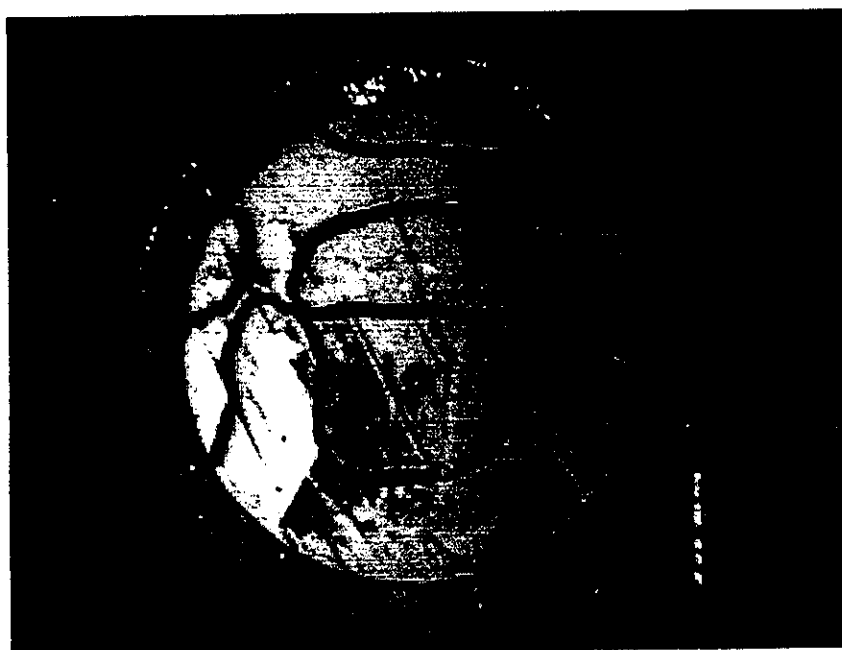
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**Figure 1e: Shell Mix #1 - Cut E**



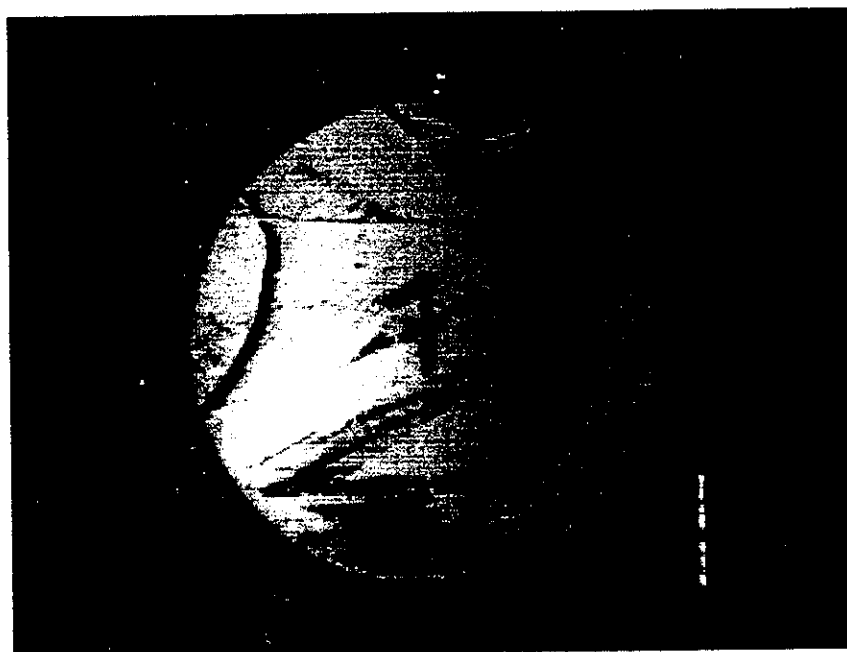
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**Figure 1f: Shell Mix #1 - Cut F**



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Figure 1g: Shell Mix #1 - Cut G



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Figure 1h: Shell Mix #1 - Cut H



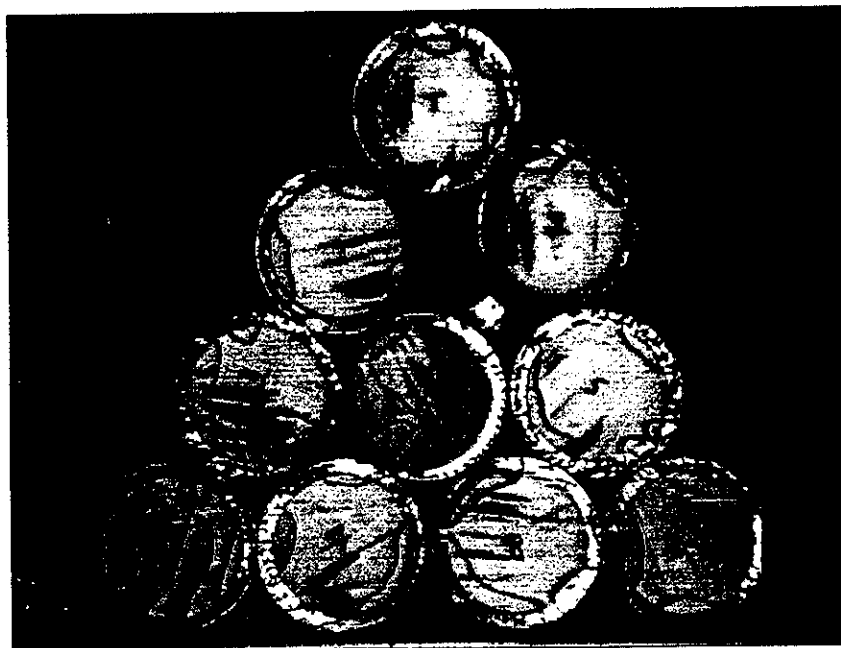
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Figure 1i: Shell Mix #1 - Cut I



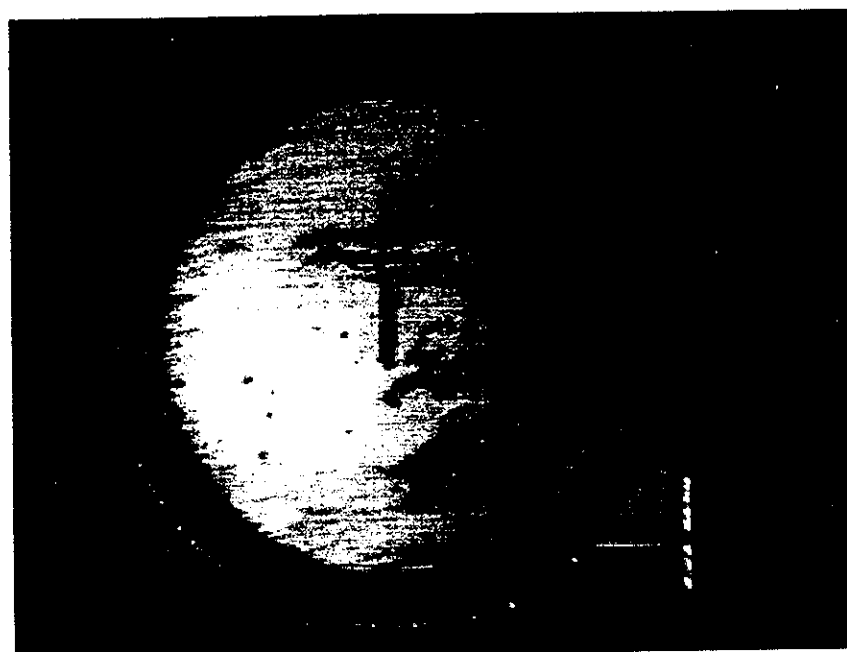
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Figure 1j: Shell Mix #1 - Cut J



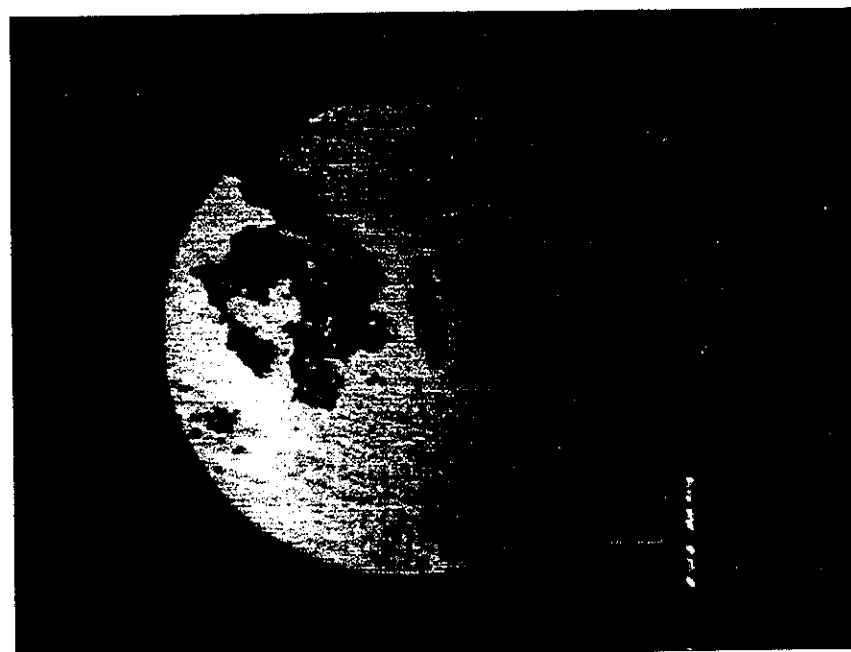
**Figure 1k - Shell Mix #1 - Cuts A through J**





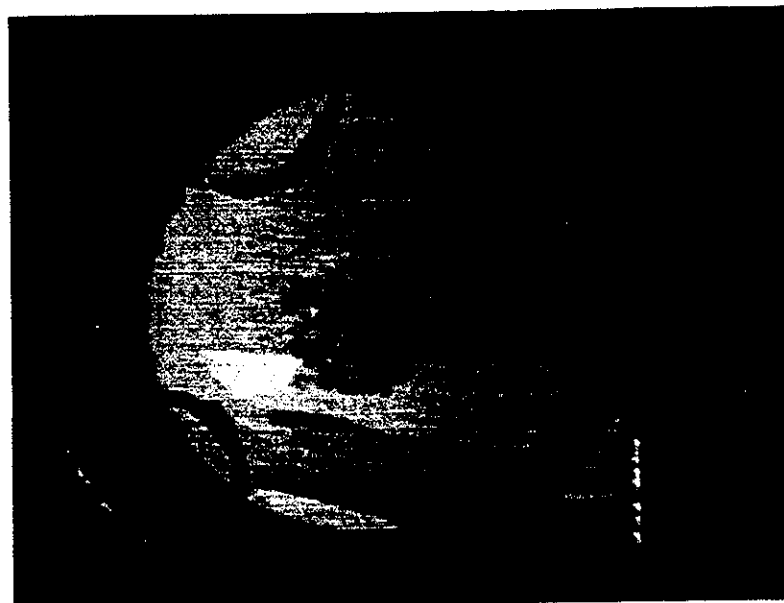
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Figure 2a: Shell Mix #2 - Cut A



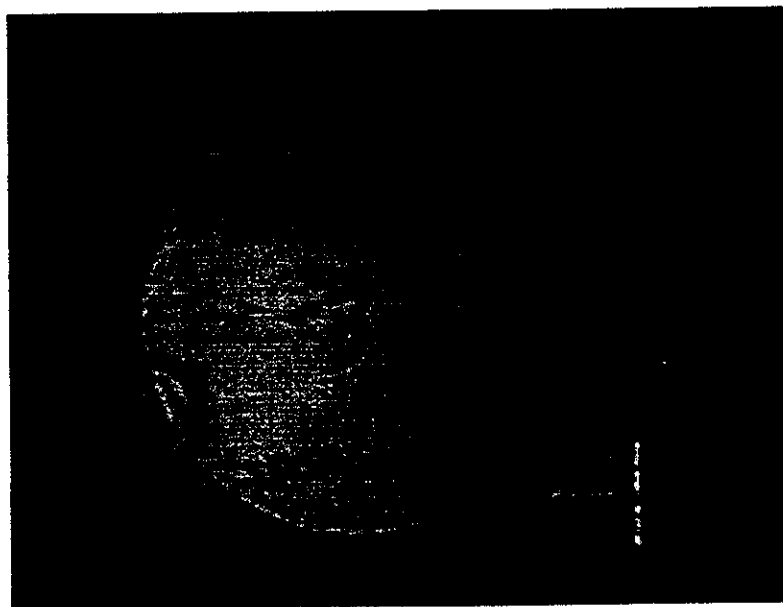
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Figure 2b: Shell Mix #2 - Cut B



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Figure 2c: Shell Mix #2 - Cut C



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Figure 2d: Shell Mix #2 - Cut D

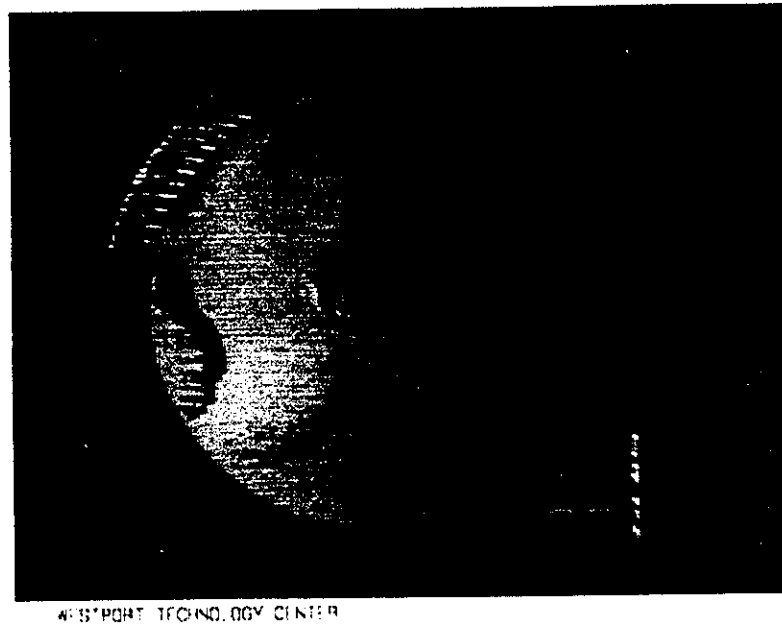
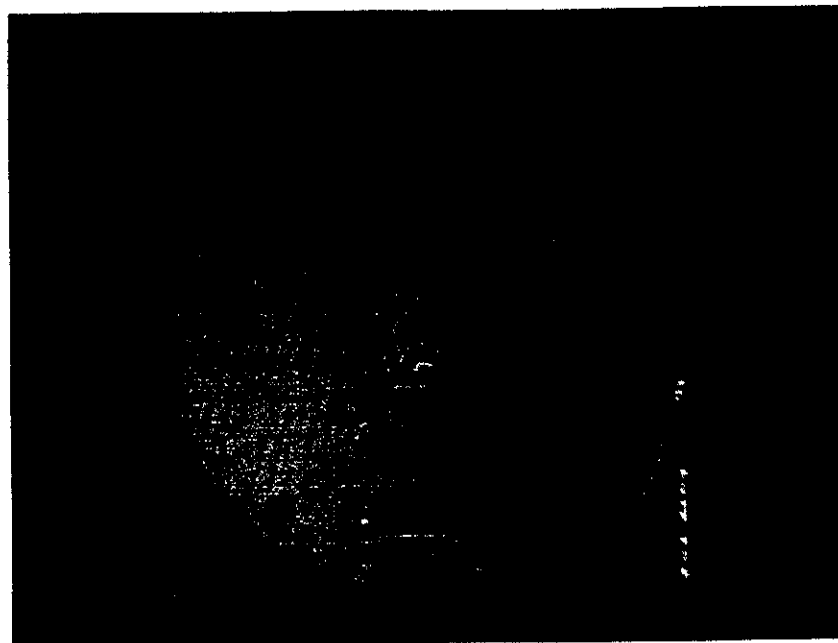


Figure 2e: Shell Mix #2 - Cut E

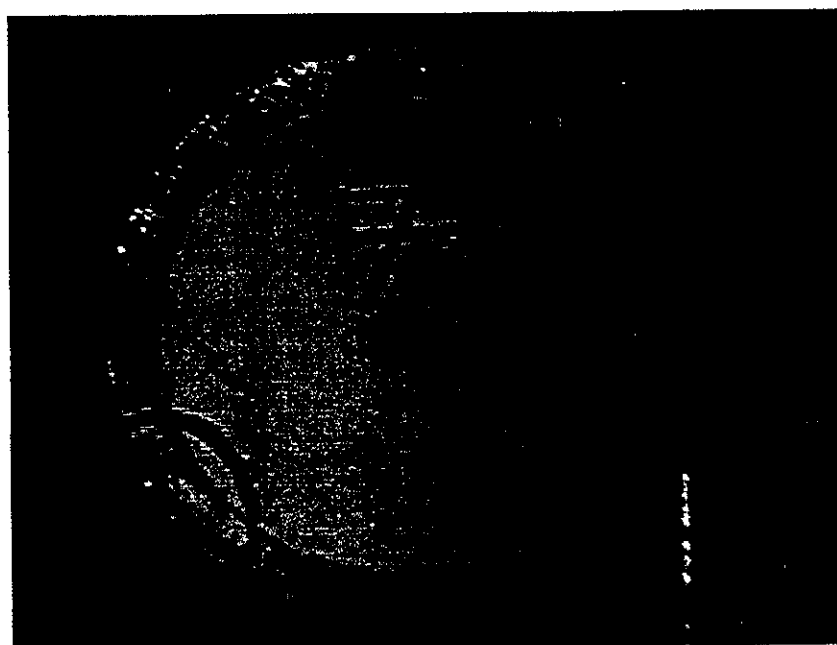


Figure 2f: Shell Mix #2 - Cut F



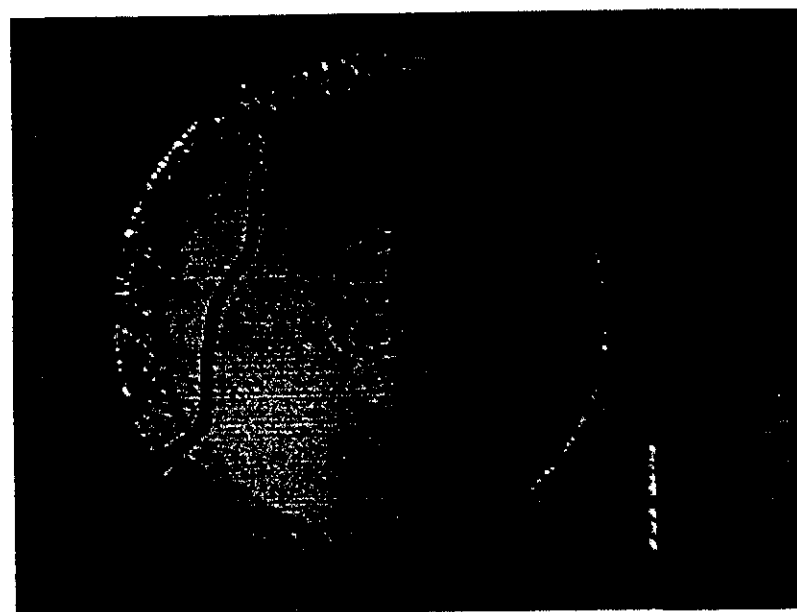
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Figure 2g: Shell Mix #2 - Cut G



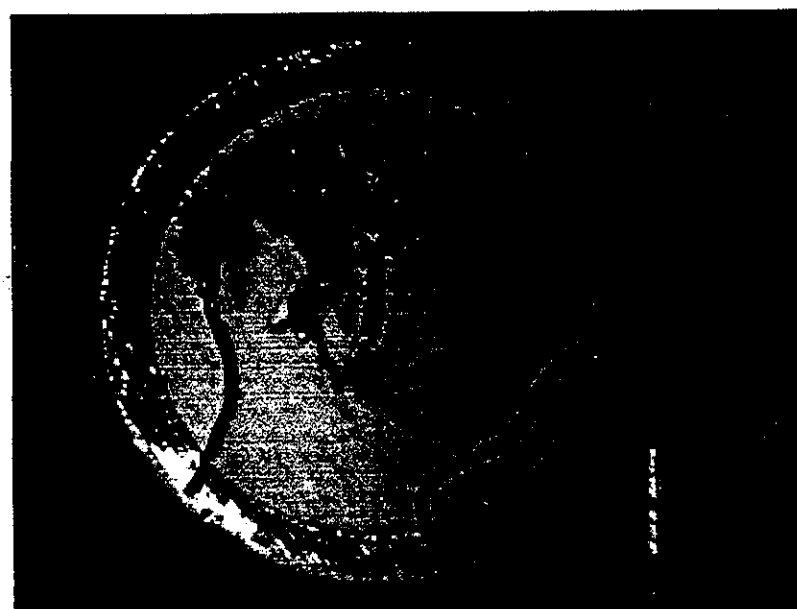
WESTPORT TECHNOLOGY CENTER

Figure 2h: Shell Mix #2 - Cut H



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Figure 2i: Shell Mix #2 - Cut I



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Figure 2j: Shell Mix #2 - Cut J

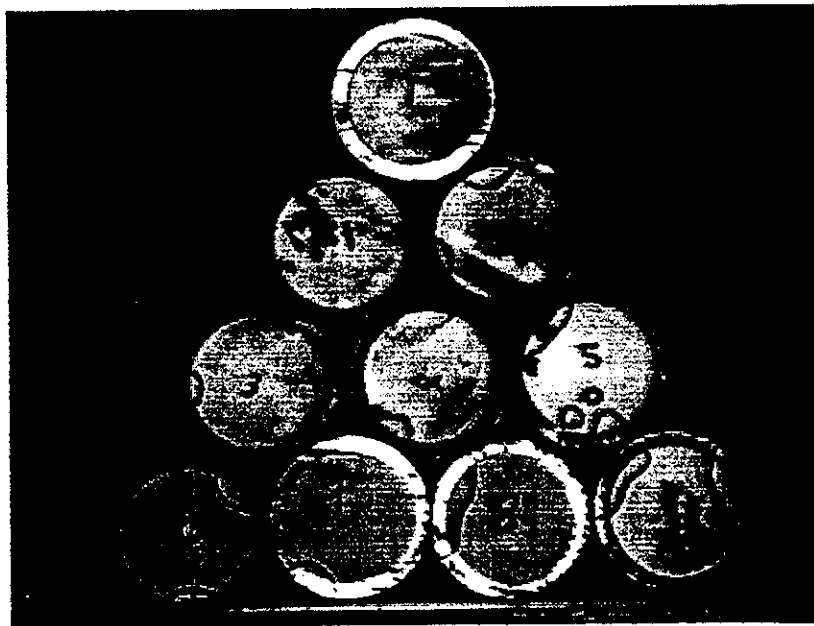
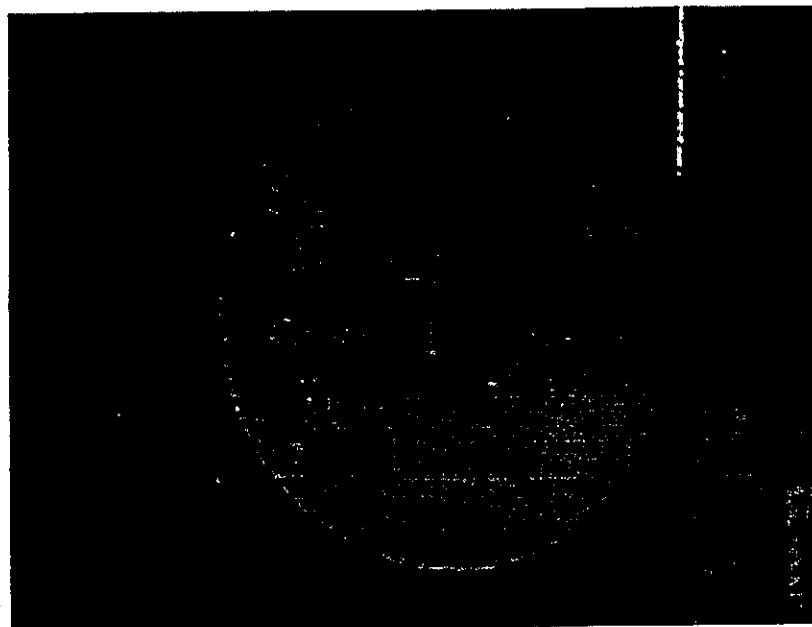
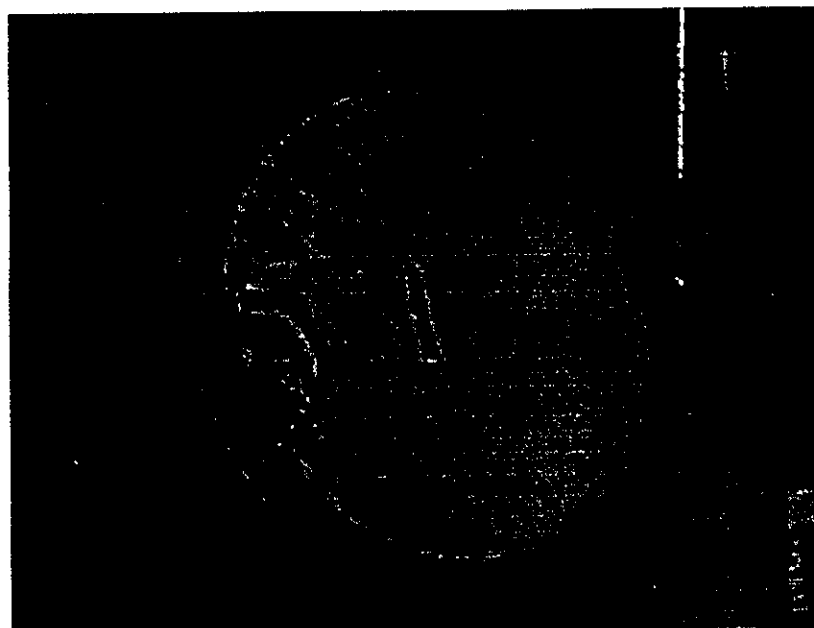


Figure 2k - Shell Mix #2 - Cuts A through J



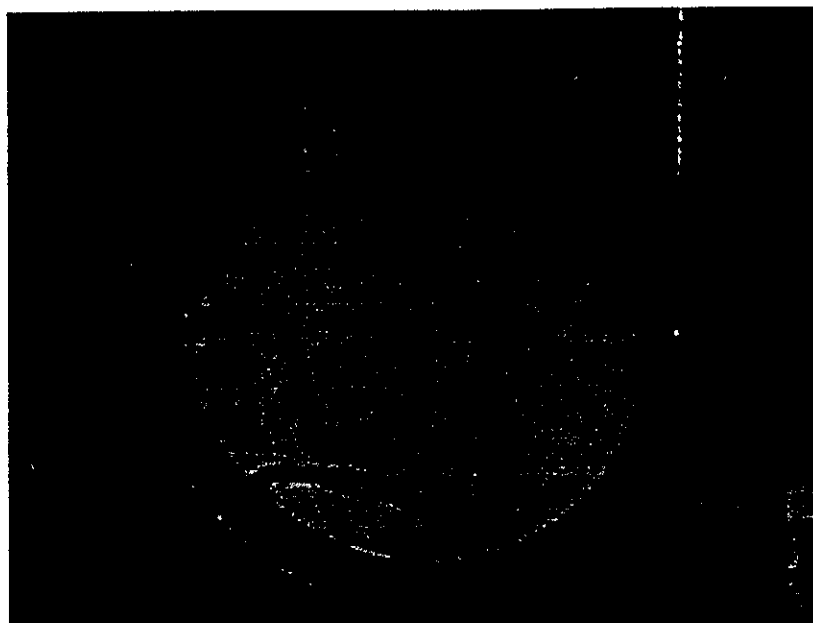
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**Figure 3a: Shell Mix #3 - Cut A**



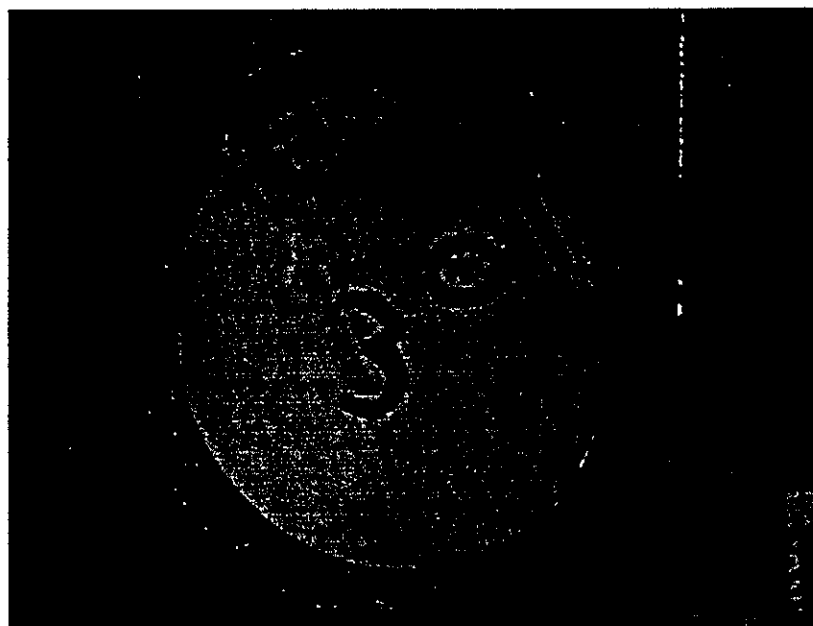
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**Figure 3b: Shell Mix #3 - Cut B**



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**Figure 3c: Shell Mix #3 - Cut C**



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**Figure 3d: Shell Mix #3 - Cut D**



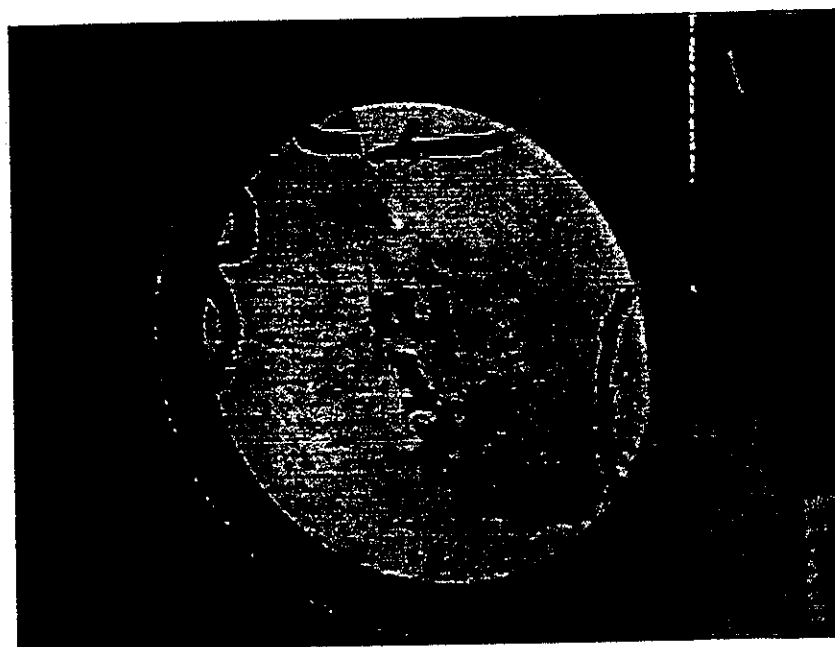


Figure 3e: Shell Mix #3 - Cut E

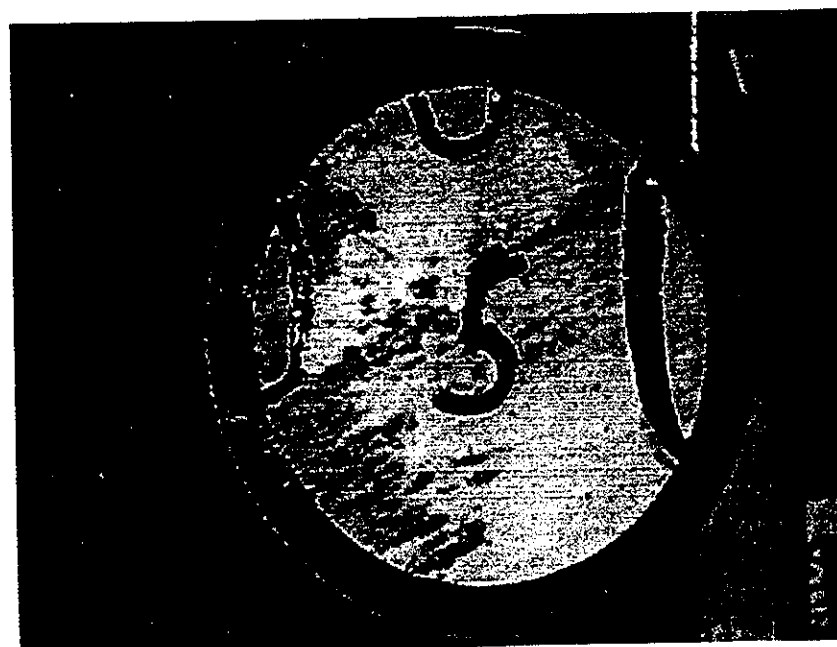
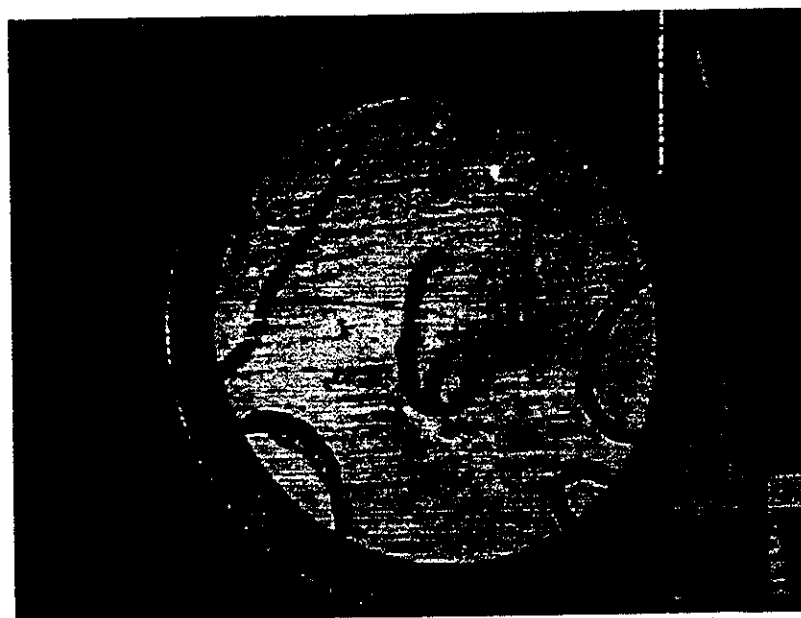
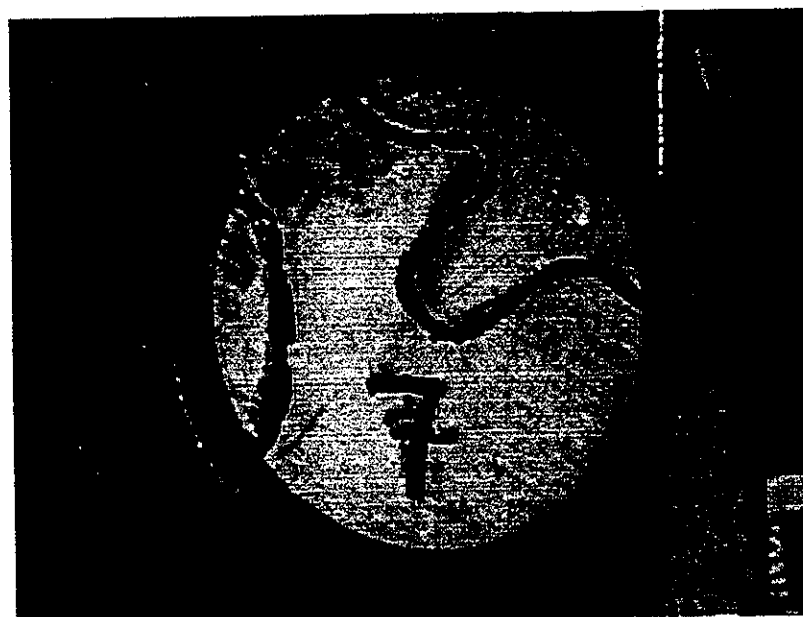


Figure 3f: Shell Mix #3 - Cut F



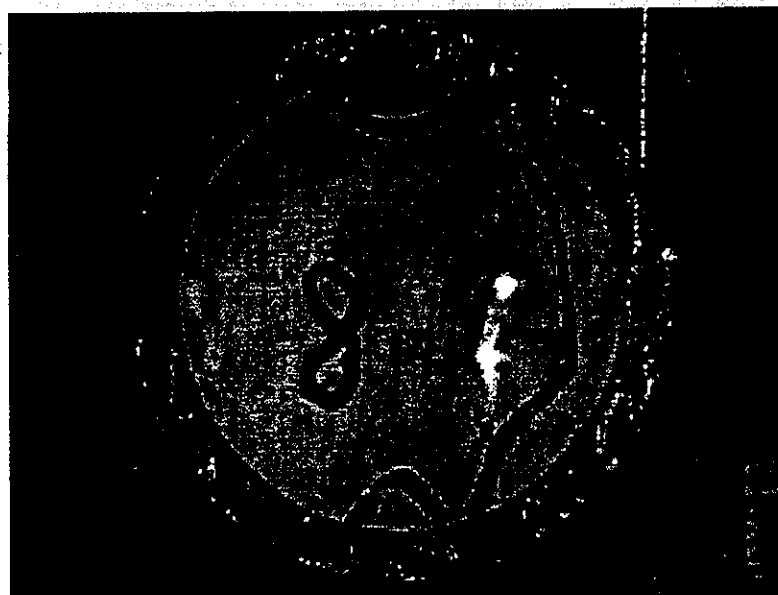
WESTPORT TECHNOLOGY CENTER

Figure 3g: Shell Mix #3 - Cut G



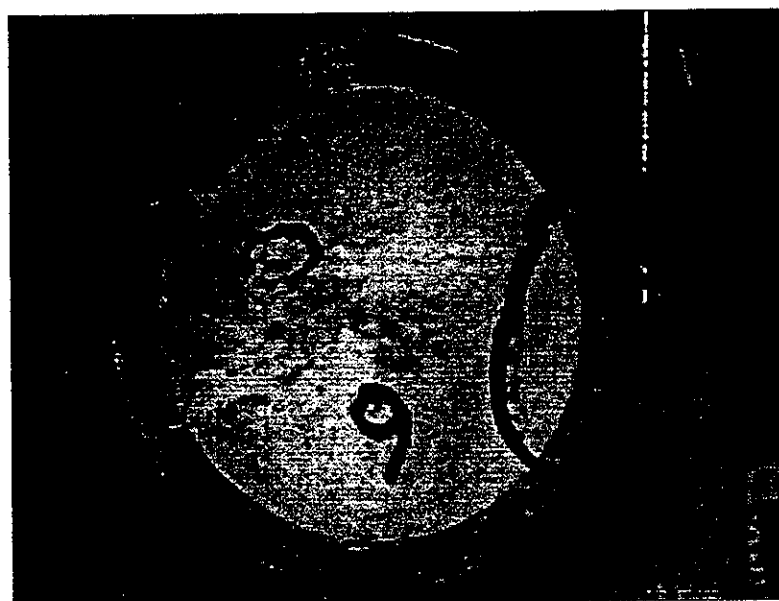
WESTPORT TECHNOLOGY CENTER

Figure 3h: Shell Mix #3 - Cut H



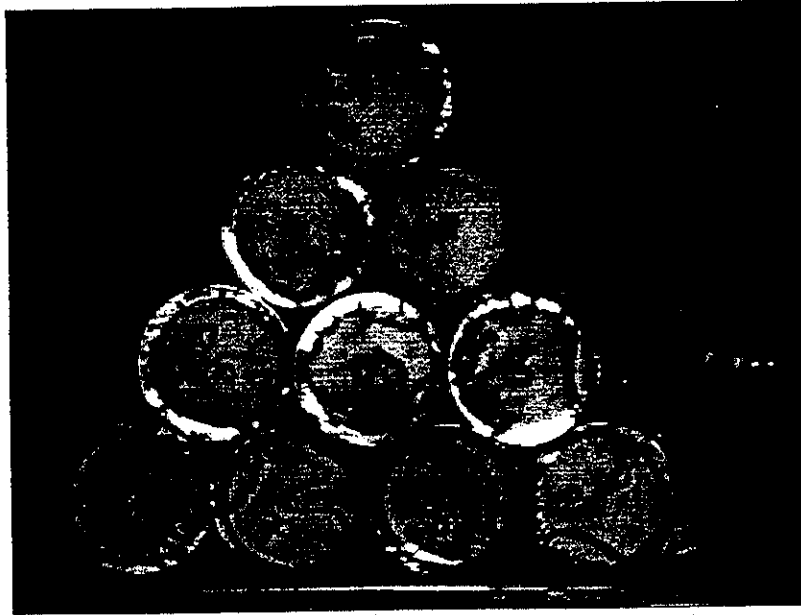
WESTPORT TECHNOLOGY CENTER

Figure 3i: Shell Mix #3 - Cut I

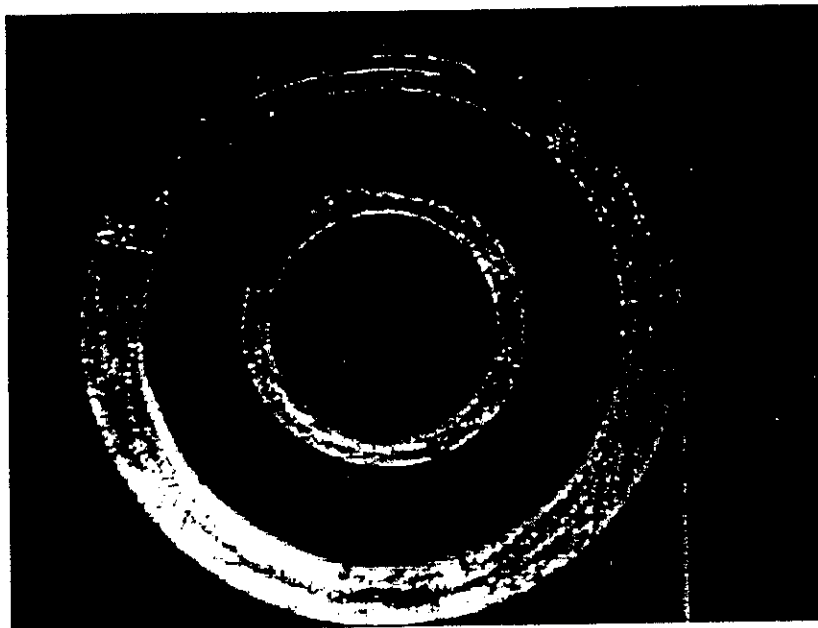


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Figure 3j: Shell Mix #3 - Cut J



**Figure 3k - Shell Mix #3 - Cuts A through J**



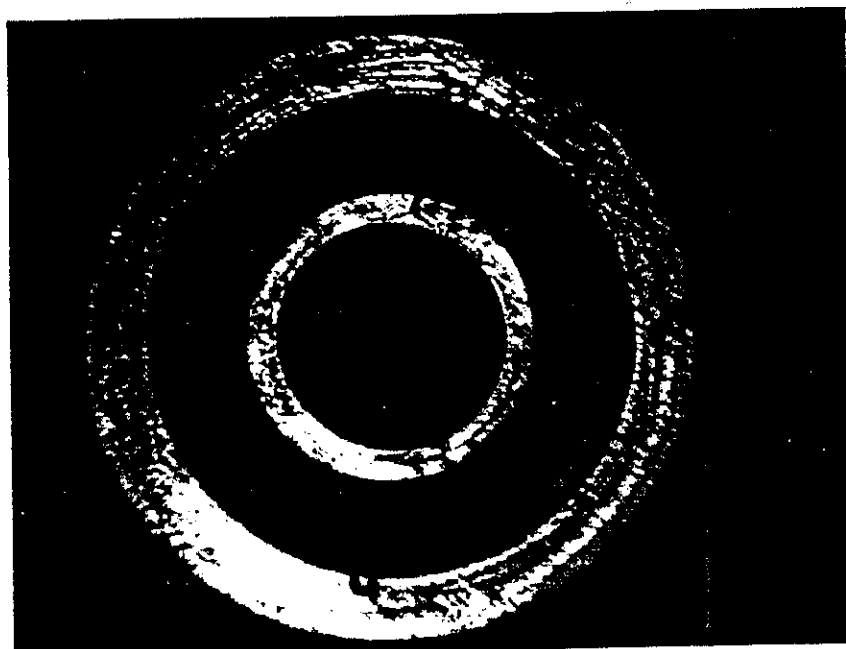
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Figure 4: Portland Cement Annular Seal



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Figure 5: Shell Mix #2 Annular Seal



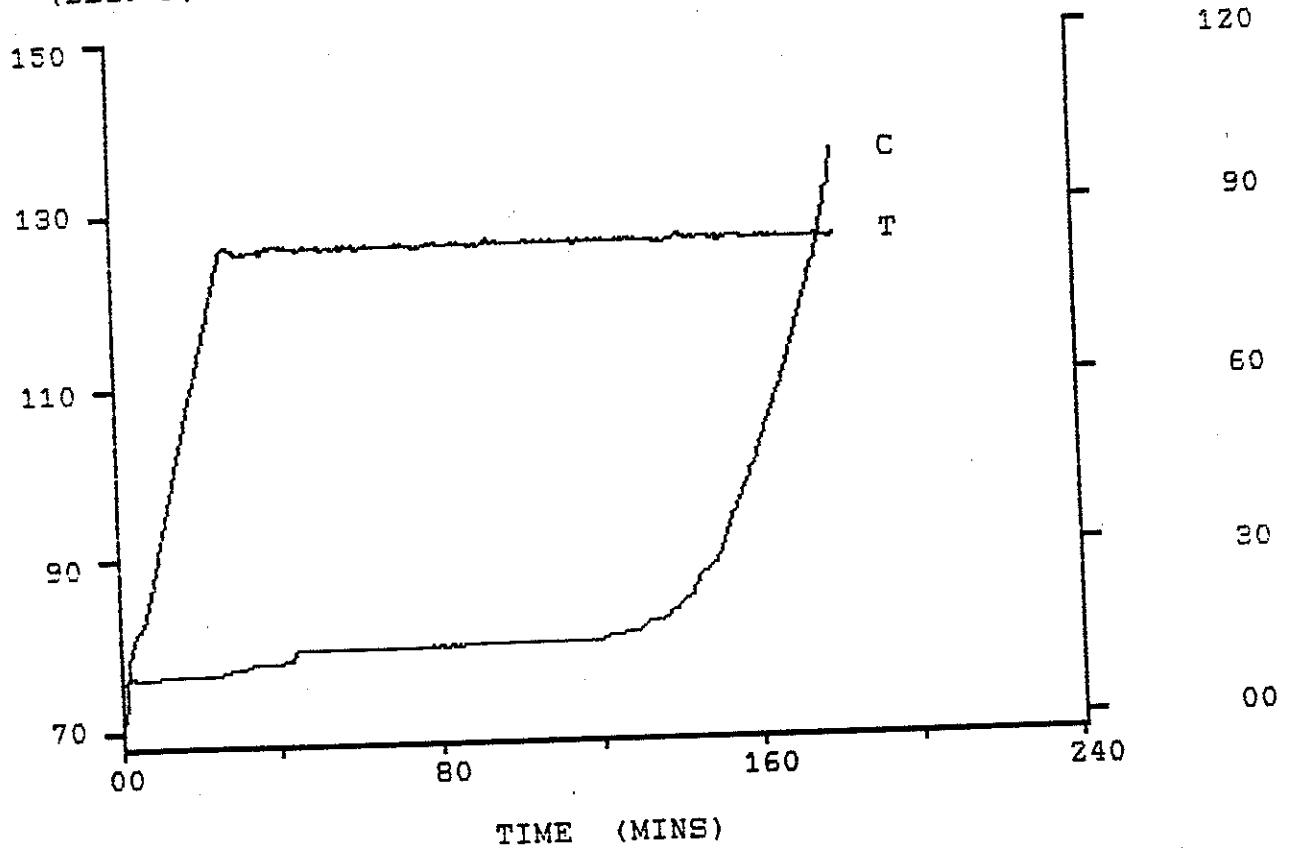
WESTPORT TECHNOLOGY CENTER

**Figure 6: Shell Mix #3 Annular Seal**

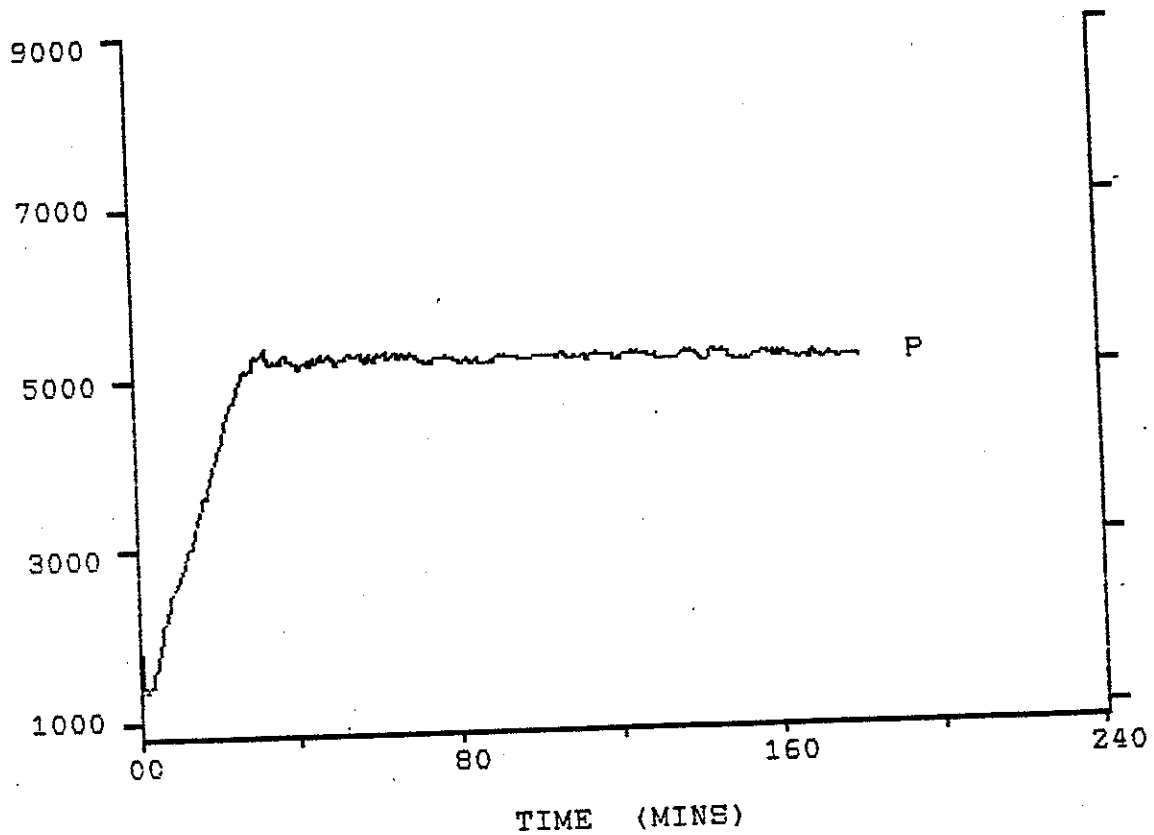
DEA-87 Phase II  
Shell Mix #2 Thickening Time  
3:01 (181') to 100 Bc

TEMPERATURE  
(DEG. F)

CONSISTENCY  
BEARDEN



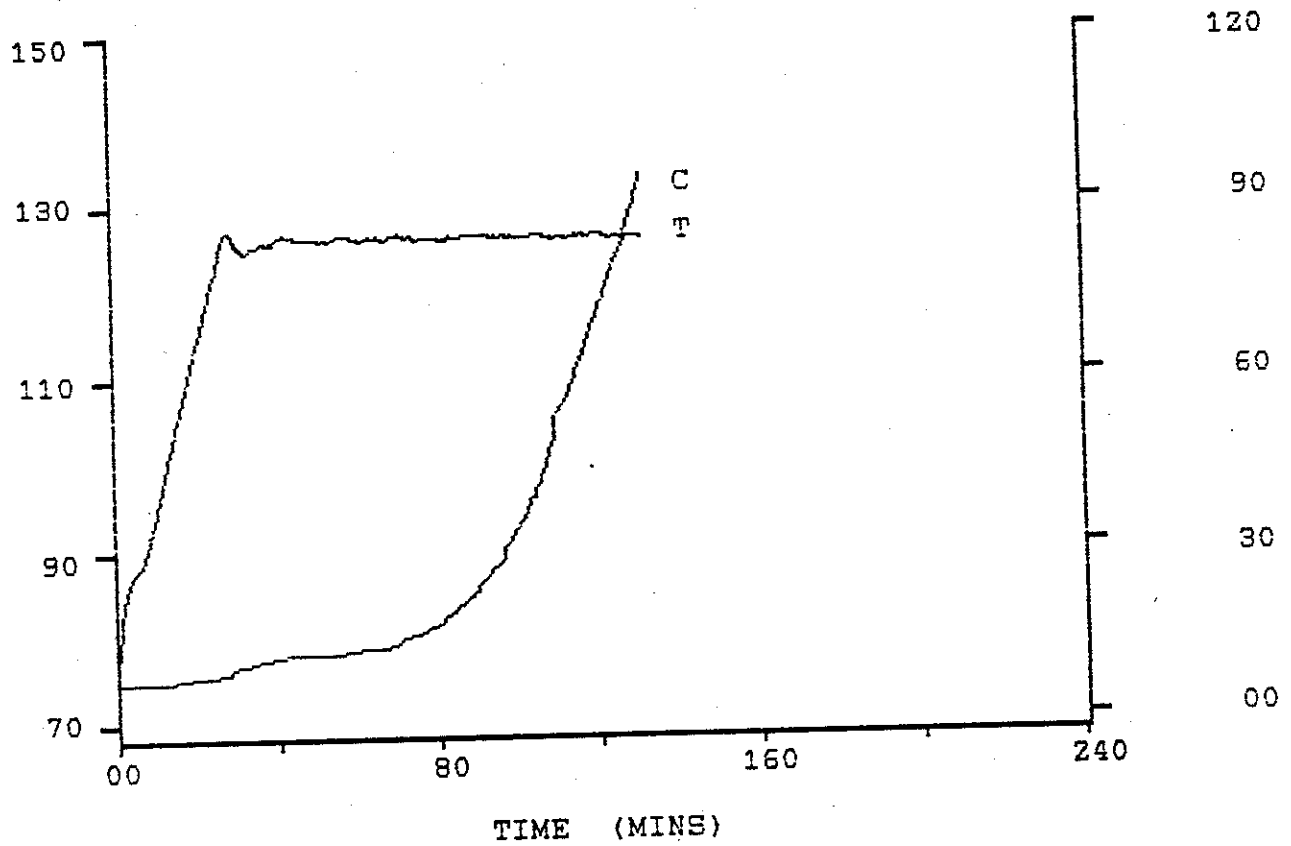
PRESSURE  
(PSI)



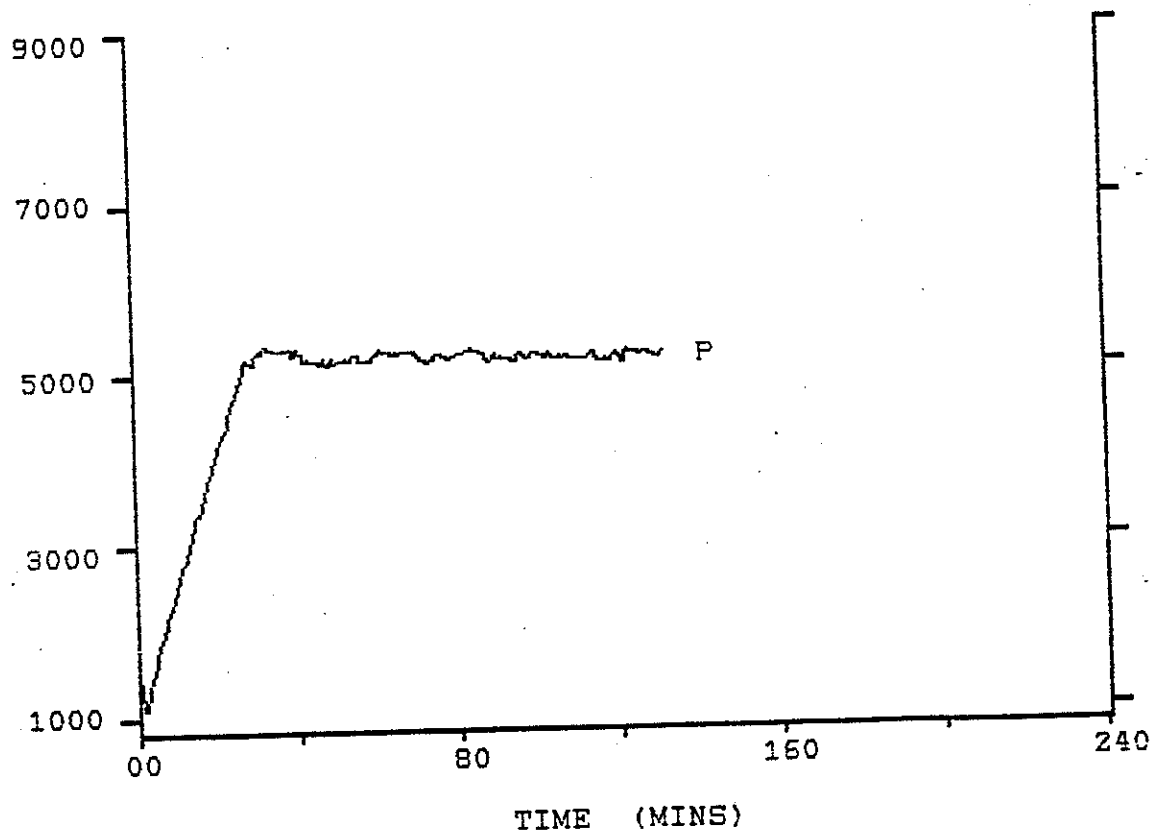
DEA-87 Phase II  
Shell Mix #3 Thickening Time  
2:12 (132') to 100 Bc

TEMPERATURE  
(DEG. F)

CONSISTENCY  
BEARDEN



PRESSURE  
(PSI)





DEA-87 Phase II

PROJECT NO.:

DATE:

PRESSURE:

TEMPERATURE:

ULTRASONIC

CEMENT ANALYZER

HALLIBURTON SERVICES

INITIAL SET: 50 @ 4:36

STRENGTH 1: 500 @ 5:02

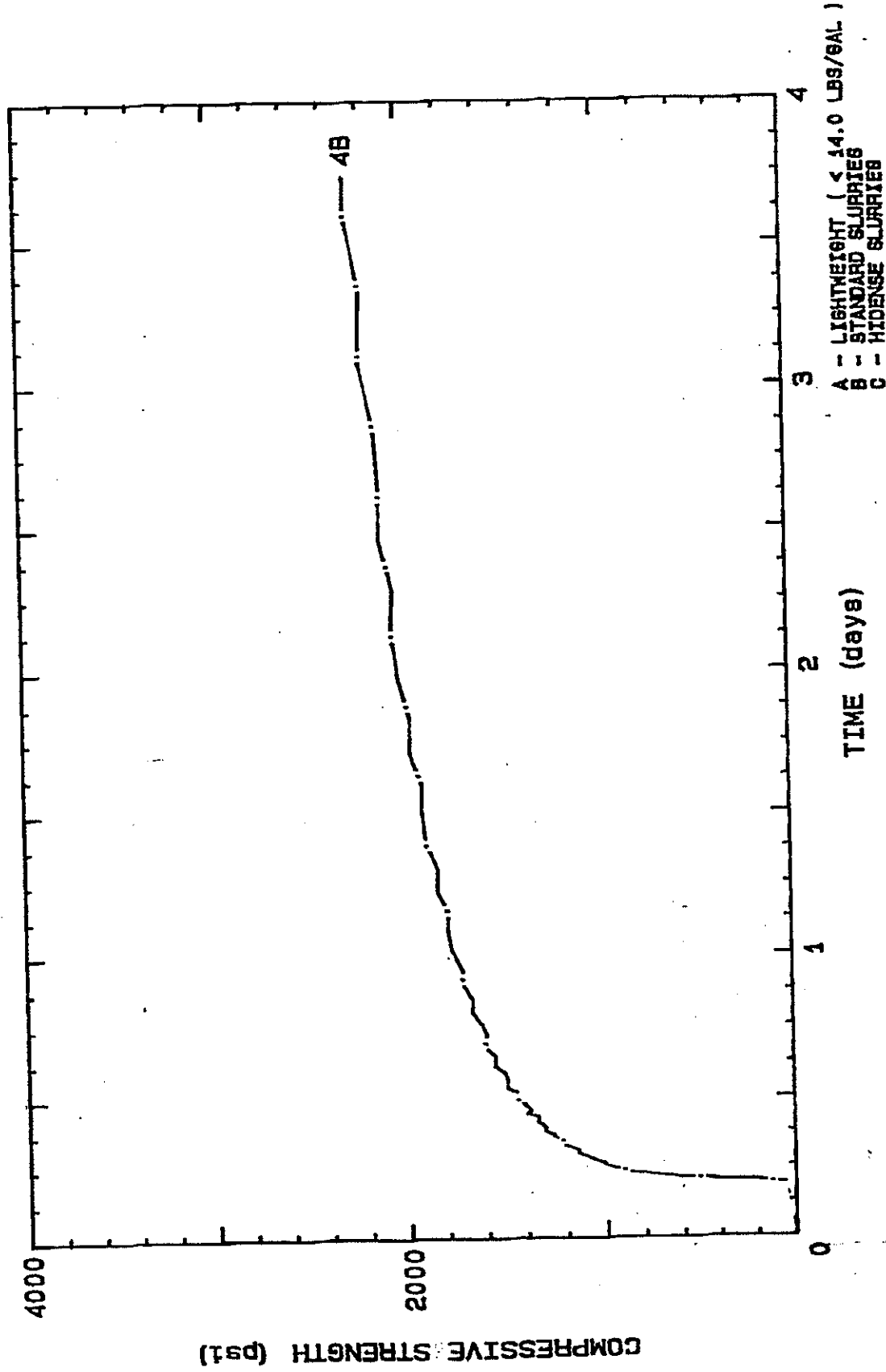
STRENGTH 2: 19999

CURR. STR.: 2274 @ 89:47

Shell Mix #1: 12.5 ppg PHPA diluted to 10.5 ppg + 5.52 lb/bbl Sodium Hydroxide

+ 13.81 lb/bbl Sodium Tripoly Phosphate + 314.3 ppb Blast Furnace Slag at 15.2 ppg

CEMENT:



PROJECT NO.: DEA-87 Phase II

DATE: 05/16/97

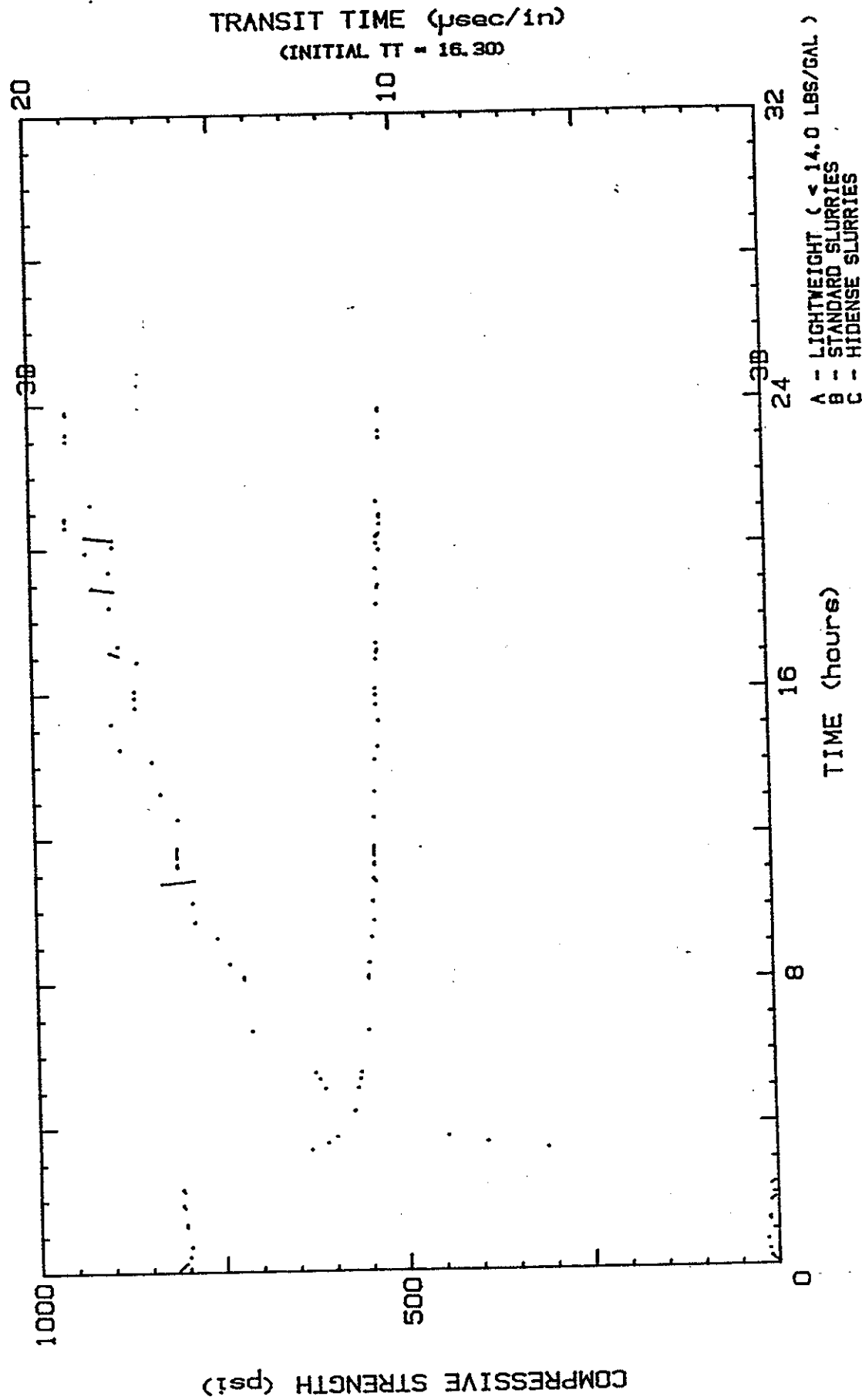
PRESSURE: 3000 PSI

TEMPERATURE: 152°F BHST

ULTRASONIC  
CEMENT ANALYZER  
WESTPORT TECHNOLOGY

INITIAL SET: 50 0 2:54  
STRENGTH 1: 500 0 3:58  
STRENGTH 2: 19999  
CURR. STR.: 950 0 24:03

CEMENT: Shell Mix #2: 12.5 ppg PHPA diluted to 10.5 ppg + 4.43 lb/bbl Sodium Hydroxide  
+ 11.07 lb/bbl Lime + 1.85 lb/bbl Dispersant + 314.3 ppb Blast Furnace Slag at 15.2 ppg



PROJECT NO.: DEA-87 Phase II  
DATE: 09/04/97  
PRESSURE: 3000 PSI  
TEMPERATURE: 152°F BHST

ULTRASONIC  
CEMENT ANALYZER  
WESTPORT TECHNOLOGY

INITIAL SET: 50 0 2:30  
STRENGTH 1: 500 0 4:02  
STRENGTH 2: 19999  
CURR. STR.: 1071 0 25:16

CEMENT: Shell Mix #3: 12.5 ppg PHPA diluted to 10.5 ppg + 4.43 lb/bbl Sodium Hydroxide  
+ 11.07 lb/bbl Sodium Carbonate + 2 gal/bbl NRJ-1428 Resin + 314.3 ppb Blast Furnace Slag at 15.2 ppg

